# THE HISTORY OF TERRA-COTTA GLAZE-FIT TESTING AND ARTIFICIAL WEATHERING METHODOLOGIES AND A COMPARATIVE TESTING PROGRAM OF THEIR IMPACT ON GLAZE FAILURE

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## 1. INTRODUCTION

Terra cotta failure is often due to glaze defects, including crazing, crawling, and spall. The makeup of both the clay body and the glaze, and manufacturing processes, are of paramount importance in the prevention of these defects. As part of the fabrication process, ceramicists often put terra-cotta wares through a variety of tests to aid in the prediction of a product's durability. Artificial weathering is a valuable technique for gaining such information.

Today there are common procedures in place published by the American Society for Testing and Materials (ASTM) and Building Research Establishment (BRE) to mimic the freeze-thaw cycles, moisture, and presence of salts that are typical in real-world conditions. When terra cotta was still a relatively new product in the early 20$^{th}$ century, however, these procedures were not standardized, and neither were techniques for terra cotta production.

This thesis explores historic tests that were utilized to improve the longevity of terra cotta as an architectural material. I assembled a chronology, and examined trends seen in testing, focusing on tests that either studied glaze fit or utilized artificial weathering. The unconventionality of some of these tests, when considered in the context of modern procedures, led me to wonder how effective they might have been. I also wondered if there could have been useful information that had been lost on today's industry over the near century since the height of terra cotta's use. Since there was not a clear connection between many of the historic and contemporary tests, it seemed likely that they could produce relatively diverse results and provide different types of information for ceramicists and conservators.

In order to better understand and ultimately evaluate these weathering techniques, I put samples from various New York City buildings through both historic and contemporary methods of artificial weathering. These tests were then evaluated for their ability to increase the possibility of glaze failure by performing surface adhesion pull-tests. Although the results of these final tests were numerically scattered, they still provided useful insight into the value of the weathering tests that had been reproduced.

## 2. MATERIAL HISTORY

### 2a. Use in America

Terra cotta was widely used in the United States as a decorative building material between 1880 and 1930. It is a manufactured material which often consisted of two parts: the main structure is a body made primarily of clay, which was then sometimes coated in a layer of slip or glaze. The clay body is comprised of clay, usually sourced locally by the manufacturer, and grog. Grog is broken pieces of previously fired terra cotta, ceramic, or brick, and is added to control shrinkage and reduce warpage in new terra-cotta units.

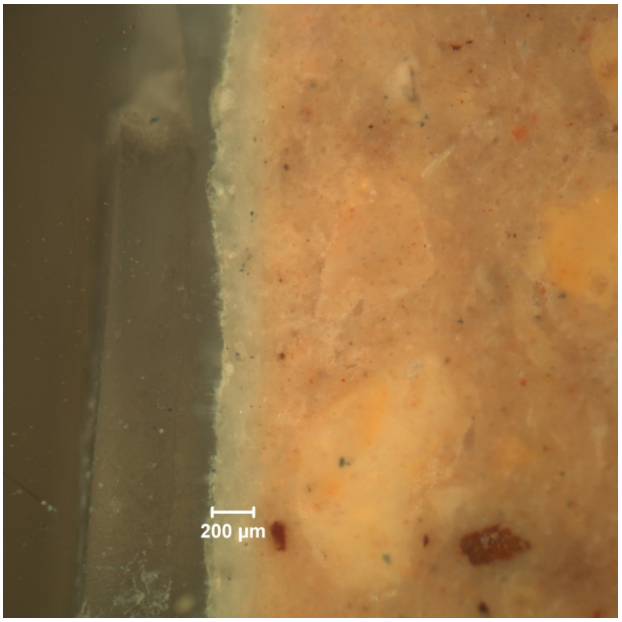


Figure 1: Cross section of terra cotta, showing glaze and clay body, which contains grog. Source: Xsusha Flandro

Clay bodies were traditionally hand-pressed into molds by factory workers, who hand-pounded the clay and added structural webs to the backs of the hollow forms.[1] Molds were created with a special ruler designed to account for terra cotta's shrinkage during firing: draftsmen used a 13

[1] Veit, Richard. "Moving Beyond the Factory Gates: The Industrial Archaeology of New Jersey's Terra Cotta Industry." *The Journal of the Society for Industrial Archeology* 25.2 (1999): 9.

inch ruler, divided into 12 “inch” segments.[2] Molds were usually constructed in plaster, and comprised of many pieces that could be disassembled face-by-face.[3] Units were left to dry in these molds for a few days, and then were taken out of the molds for an additional two to three days. Imperfections were smoothed and finished by hand, and the piece was then brought to a steam-heated drying room.[4]

Once properly dried, pieces were coated with slip (a watered-down version of clay) or glaze (a glassy, silica-based coating) by brushing or spraying.[5] Although dipping is a common technique used to apply glaze in an artistic ceramic setting, due to the size and weight of architectural terra cotta units, it was not common in the industry.[6] Terra-cotta units were then fired in a kiln, a process which could take one to two weeks. The temperature began to rise slowly, which allowed for the complete driving out of any water remaining in the units- this period was called “the water smoking period,” “steaming,” or “going through the sweat.”[7] Once all water was removed, the temperature was raised more quickly to 2,200-2,500°F, where it remained for 90-150 hours.[8] The kiln was then cooled gradually, preventing the glaze defects or unit cracking that could come with rapid cooling.

Terra cotta quickly became a popular building material for many reasons. Firstly, it drove down the per-unit cost of decorative elements, since the molds could be reused. The initial mold carving, done by hand, only had to be carried out once by a sculptor, and the mold could then

[2] Tunick, Susan. *Terra-Cotta Skyline: New York’s Architectural Ornament* (Princeton: Princeton Architectural Press, 1996), 33-34.
[3] Tunick, Susan. *Terra Cotta- Don’t Take it for Granite: 3 Walks in New York City Neighborhoods*. (New York: The Friends of Terra Cotta Press, 1995), 3-4.
[4] Veit, 9.
[5] Tunick. *Terra Cotta- Don’t Take it for Granite: 3 Walks in New York City Neighborhoods*, 3-4.
[6] Tunick, Susan. "Terra-Cotta Manufacturing." Personal interview. 18 Apr. 2011.
[7] Materio, Frank G., Elizabeth A. Bede, and Albert Tagle. *An Evaluation of the Effects of Cleaning Methods on Unglazed Architectural Terra Cotta: With Notes on the Manufacture and Characterization of Nineteenth-Century American Production*. (Philadelphia: Architectural Conservation Laboratory, Graduate School of Fine Arts, University of Pennsylvania, 1995), 29.
[8] Veit, 10.

be repeatedly used over a façade or even on multiple buildings with consistently crisp results.[9] Secondly, terra cotta was thought to be a long-lasting, damage-proof material. Promotional material of the time described terra cotta as indestructible, self-cleaning, and non-absorbent.[10] Especially when a piece was glazed, it was thought that this vitrified, glass-like layer would protect the unit from all water-related damage.[11]


Figure 2: Catalog representation of polychrome terra cotta, Source: *Atlantic Polychrome Terra Cotta*, 1917.

The primary goal of this thesis is not to detail the history of terra cotta; it is understood that the reader has a basic knowledge of the origin and use of the material. Further information on the history of terra cotta can be found in the introductory chapters of the following theses and dissertation:

- Allen, Daniel. *An Evaluation of Mortars for the Plastic Repair of Architectural Terra Cotta*. Thesis. Columbia University, 1993.
- Flandro, Xsusha Carlyann. *Glazed and Confused: Exposing the Mysteries of Glazed Architectural Terra Cotta*. Thesis. Columbia University, 2009.

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[9] Tiller, De Teel Patterson. "The Preservation of Historic Glazed Architectural Terra-Cotta." *Preservation Briefs*. National Park Service. <http://www.nps.gov/hps/tps/briefs/brief07.htm>.
[10] Ibid.
[11] Ibid.

- Prudon, Theodore H.M. *Architectural Terra Cotta and Ceramic Veneer in the United States Prior to World War II: A History of its Development and an Analysis of its Deterioration Problems and Possible Repair Methodologies*. Diss. Columbia University, 1981.

**2b. Material Properties & Defects**

In many ways, terra cotta is used and behaves similarly to stone and brick veneers. It is made of block-shaped units, which are set into a façade and tied back to supporting structure. In fact, early uses of terra cotta often sought to replicate and imitate stones of all colors and textures.[12] Just like other cladding systems, terra-cotta units are held in place by anchoring systems and/or brick backfill; the anchors were often made of ferrous metals. Although early literature professed terra cotta to be non-absorbent,[13] this is not the case; porous mortars, improper flashing, and glaze defects can all allow for water infiltration. When iron rusts, it expands, and this can rupture a terra-cotta unit, causing pieces to fall off the building.

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[12] "Terra Cotta- Ancient and Modern." *The Clay-Worker* 32 (1899): 191-192.
[13] Tiller.

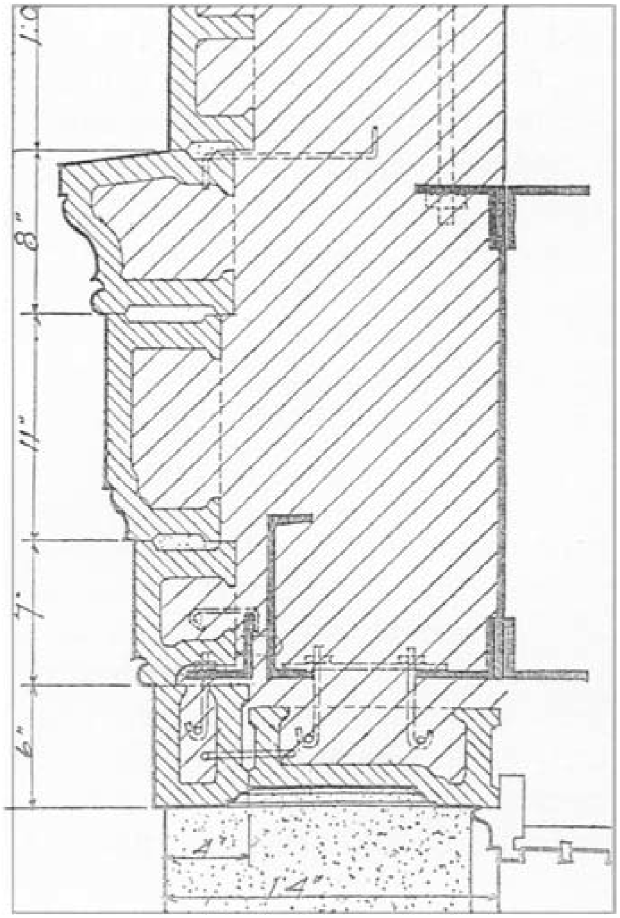


Figure 3: Typical construction detail of architectural terra-cotta ornament, showing anchoring system. Source: National Terra Cotta Society, *Architectural Terra Cotta: Standard Construction*, 1914.

However, terra cotta poses a unique set of deterioration issues not encountered with traditional masonry materials for two reasons. Firstly, it is a synthetic product, and is manufactured with natural materials. Secondly, it involves the duality of two very different components, the clay body and the glaze, which are united in such a way that they must perform similarly in order to be successful as a whole unit. The juxtaposition of the glassy glaze and the porous clay body, which are made of different materials, is often a source of problems that are unique to terra cotta as a building material.

Water absorption can promote further problems within a terra-cotta unit. If water from mortars or another source, including the clay itself, contains salts, when the salts crystalize, they will expand within the pores of the clay body. This pressure can rupture the glaze and clay body, causing a small portion of the glaze to pop off of the surface, often bringing part of the clay body with it. This type of damage is referred to as “spalling”, with the void on the unit being called a “spall.” In addition to the visual effect this has, the piece is then even more susceptible to water damage, since the clay body of glazed architectural terra cotta was never designed to be exposed. Spalls can also be furthered by biological growth underneath the surface of the glaze, perpetuated by the presence of water. However, if biological growth is present under the glaze, then it is an indication that there are already glaze adhesion problems that would give room for this growth.

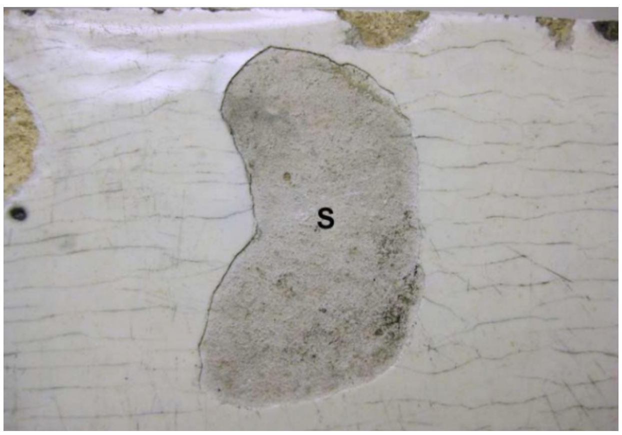
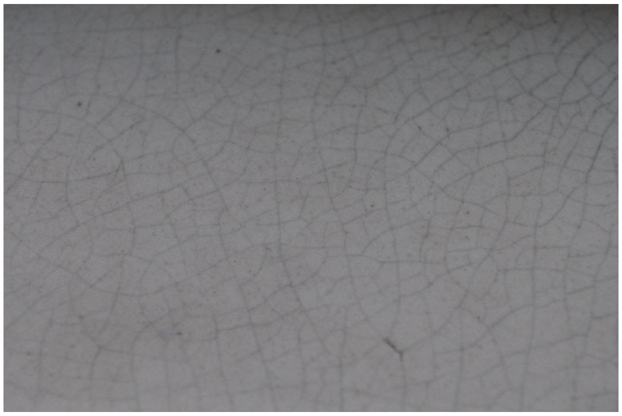



Figure 4 (left): An example of glaze spall. Source: Joan Berkowitz
Figure 5 (right): Glaze spall, furthered by biological growth beneath the glaze surface. Source: Joan Berkowitz

Crazing is another glaze defect common in terra cotta. Glazes are, ideally, a vitrified surface coating on the terra cotta’s clay-body surface. They are less likely to expand due to moisture, since they do not absorb water nearly as much as the porous clay body- any expansion that may occur in the glaze is more likely due to thermal changes. When a clay body expands and contracts at a rate different from the glaze, this can cause hairline fractures within the glaze, called crazing. Crazing can happen during the unit’s initial cooling period in the kiln, during initial exposure periods during storage at the factory, or it can occur over the course of the material’s lifetime and use on a building. It is often difficult to tell the age of crazing cracks in a glaze. Xsusha Flandro’s 2009 thesis proved that these types of defects in a glaze surface, although visually unobtrusive, do allow for minimal increased water absorption through the glaze into the clay body. Sometimes, however, the visual appearance of crazing was desirable, and ceramicists purposely created the cracks.[14]



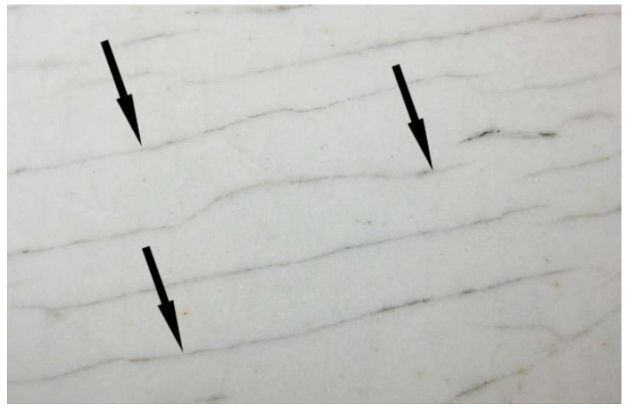


Figure 6 (left): Crazing on a glazed surface in a typical web-like pattern
Figure 7 (right): Crazing lines running parallel to one another (cause of pattern is unknown). Source: Joan Berkowitz

Shivering, another form of glaze loss, may appear similar to spalling, in that it also entails chip-like pieces of glaze separating from the terra-cotta unit. However, there is a difference between these types of defects- shivering does not involve the loss of clay body, but rather only glaze loss, as if it "peels" off the clay surface. It is caused by excess amounts of silica and/or salts in the clay body, and is exacerbated by compression forced upon the unit.[15] Some writing also describes shivering as glaze peeling.

[14] Flandro, Xsusha Carlyann. "Answers to Questions." Message to the author. 31 Jan. 2012. E-mail.
[15] Schurecht, H.G and G.R. Pole. "Method of Measuring Strains between Glazes and Ceramic Bodies." *Journal of the American Ceramic Society* 13.6 (1930).


Figure 8: Peeled glaze, without clay body loss. Source: Xsusha Flandro

Crawling is similar to crazing in that involves separation of the glaze from the body, but instead of cracking during final cooling or weathering, the glaze contracts during the actual firing or immediately following cooling period. This results in unglazed portions of clay body which are surrounded by coalesced glaze, rather than sharply fragmented edges.


Figure 9: Crawling on a glazed surface

## 3. HISTORIC LITERATURE SURVEY

I began this thesis as a study in the evolution of terra-cotta clay bodies and the effect of changing techniques on glaze fit and ultimate unit performance. Manufacturers rarely published their trade secrets of composition and techniques, and so this led me to a search through historic literature for tests that may have ultimately influenced methodologies that were used in practice. I found that glaze defects were frequently discussed and many tests sought to remedy them. Today it is common knowledge that weathering and the effect of natural forces can degrade a terra-cotta unit over the passage of time, but this is an idea that was only sometimes discussed in literature of the 1920s and '30s. The following is a survey that I conducted of tests that were performed in an attempt to improve terra cotta as a building material.

### 3a. Trends in Testing

In carrying out this survey, articles from the *Journal of the American Ceramic Society* from 1918 to 1930 were consulted. Due to the scope and schedule of this thesis, surveying all literature was simply not feasible during the given time. This journal, specifically, was chosen over other available literature because of its wide recognition as a preeminent source for ceramic and terra cotta information; there was a consistent wealth of testing information that was more prominent, and specifically related to glazed terra cotta, than was found in other sources. This is not to say that these other sources do not also contain valuable information; recommendations for further research can be found in the conclusion of this thesis. The articles examined in this study have been cataloged, and specific aspects of terra cotta experimentation, techniques employed, and sought-after traits noted. This data has been arranged in a table, making the occurrences of all tests and ideas discussed in the articles easily locatable. The following were common trends found in the literature:

- Testing for the impact of glaze and/or slip content: Tested 9 times
- Grog size: Tested 7 times, discussed an additional 3 times
- Clay body additives: Tested 7 times, discussed an additional 1 time
- Clay type: Tested 6 times
- Compressive strength: Tested 5 times, discussed an additional 1 time
- Clay body mix ratios: Tested 4 times, discussed an additional 2 times
- Clay content present in glazes: Tested 4 times
- Absorption: Tested 4 times
- Grog materiality: Tested 3 times, discussed an additional 1 time
- Glaze additives: Tested 2 times
- Ink staining: Tested 2 times
- Clay preparation: Discussed 2 times

In addition to these qualities, it was noted when weathering techniques were employed:

- Natural weathering: Tested 4 times
- Artificial freeze-thaw weathering: Tested 2 times
- Artificial weathering with an autoclave: Tested 2 times
- Artificial salt bath weathering: Tested 1 time
- Artificial weathering via quenching: Tested 1 time

The following table includes date, title, author, and types of testing. Discussion of these trends and the conclusions they drew follow. The reader should note that many terms that come up in this survey may not be commonly known; a glossary is included in the appendix.

| Journal* | Volume | Issue | Date | Title | Author | Natural Weathering | Artificial: Freeze Thaw | Artificial: Autoclave | Artificial: Salt Bath | Artificial: Quenching | Discussion Only | Glaze/Slip Content | Grog Size | Clay Additives | Clay Type | Compressive Strength | Mix Ratios | Glaze Content: Clay | Absorption | Grog Material | Glaze Additives | Ink Staining | Clay Prep |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| TACS | 2 | | Feb 1900 | Note on the Relation between the Tensile Strength of Clay Mixtures and the Size of the Grains of their Non-Plastic Constituents | Edward Orton, Jr. | | | | | | | | x | | | | | | | | | | |
| TACS | 2 | | Feb 1900 | Shivering and Crazing | Henry R. Griffen | | | | | | | | | x | | | | | | | | | |
| TACS | 4 | | Feb 1902 | Crystallized Glazes | William H. Zimmer | | | | | | | | | | | | | | | | | | |
| TACS | 4 | | Feb 1902 | Stoneware Glazes | Ross C. Purdy | | | | | | | x | | | | | | | | | | | |
| TACS | 4 | | Feb 1902 | Note on Fritting | Stanley G. Burt | | | | | | | x | | | | | | | | | | | |
| TACS | 4 | | Feb 1902 | Bacterial Growth as a Factor in Ageing Clay Mixtures | Edward C. Stover | | | | | | | | | | | | | | | | | | |
| TACS | 4 | | Feb 1902 | The Relation between the Constitution of a Clay, and its Ability to Take a Good Salt Glaze | Lawrence E. Barringer | | | | | | | x | | | x | | | | | | | | |
| TACS | 4 | | Feb 1902 | The Fluxing Power of Mica in Ceramic Bodies | Ray Thomas Stull | | | | | | | | | x | | | | | | | | | |
| TACS | 4 | | Feb 1902 | Notes on the Development of Greens from Cupric Oxide in Glazes | Francis W. Walker | | | | | | | | | | | | | | | | x | | |
| TACS | 5 | | Feb 1903 | The Work of the Ceramic Engineer in the Brick-Making Industry | Willard D. Richardson | | | | | | x | | | | | | | | | | | | |
| JACS | 1 | 3 | Mar 1918 | The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies | R.H. Minton | x | x | | x | | | | | | | x | | | x | x | | | |
| JACS | 1 | 5 | May 1918 | Polychrome Decoration of Terra Cotta with Soluble Metallic Salts | Hewitt Wilson | | | | | | | | | | | | | | | | | | |
| JACS | 1 | 9 | Sep 1918 | An Unusual Cause of Spalling of Sewer Pipe | Cullen W. Parmelee | | | | | | x | | | | | | | | | | | | |
| JACS | 1 | 10 | Oct 1918 | Notes on Sagger Clays and Mixtures | G.H. Brown | | | | | | x | | | | | x | x | | | | | | |
| JACS | 2 | 5 | May 1919 | Professional Divisions | n/a | | | | | | x | | | | | | | | | | | | |
| JACS | 3 | 1 | Jan 1920 | Some Data on the Development of Terra Cotta Glazes | E.C. Hill | | | | | | | x | | | | | | x | | | x | | |
| JACS | 3 | 2 | Feb 1920 | Notes on Terra Cotta Slips with Reference to the Use of Asbestos and Chlorite Mica | Hewitt Wilson | | | | | | | x | x | | | | | | | | | x | |
| JACS | 3 | 4 | Apr 1920 | A Satisfactory Method of Using Barium Hydrate in Terra Cotta Bodies | M.E. Gates | | | | | | | | | x | | | | | | | | | |
| JACS | 3 | 5 | May 1920 | The Effect of Variation of the Size of the Grog in Terra Cotta Bodies | R.L. Clare and D.F. Albery | x | | | | | | | x | | | | | | | | | | |
| JACS | 3 | 6 | Jun 1920 | The Composition of Kiln Gasses and Their Effect on Terra Cotta Glazes and Colors | F.B. Ortman | | | | | | | | | | | | | | | | | | |
| JACS | 4 | 1 | Jan 1921 | The Effect of Glaze Composition on the Crazing of Terra Cotta | E.C. Hill | | | | | | | x | | | | | | | | | | | |
| JACS | 4 | 6 | Jun 1921 | The Value of Ageing the Terra Cotta Body | R.L. Clare and R.N. Long | | | | | | | | | | | | | | | | | | |

| Journal* | Volume | Issue | Date | Title | Author | Natural Weathering | Artificial: Freeze Thaw | Artificial: Autoclave | Artificial: Salt Bath | Artificial: Quenching | Discussion Only | Glaze/Slip Content | Grog Size | Clay Additives | Clay Type | Compressive Strength | Mix Ratios | Glaze Content: Clay | Absorption | Grog Material | Glaze Additives | Ink Staining | Clay Prep |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| JACS | 5 | 6 | Jun 1922 | Some Experiments on the Firecracking of Terra Cotta | E.C. Hill | x | | | | | | | x | | x | x | | | | | | | |
| JACS | 5 | 8 | Aug 1922 | Notes on Shivering of Terra Cotta | John L. Carruthers | | | | | | | | | x | x | | | | | x | | | |
| JACS | 5 | 8 | Aug 1922 | Soluble Salts and Clay Wares | Cullen W. Parmelee | | | | | | | | | x | | | | | | | | | |
| JACS | 5 | 10 | Oct 1922 | Discussion on 'Some Experiments on the Fire Cracking of Terra Cotta' | n/a | | | | | | x | | | | | | | | | | | | |
| JACS | 5 | 10 | Oct 1922 | Terra Cotta Problems Suggested for Discussion and Investigation | C.W. Hill | | | | | | x | | x | x | | | x | | | | | | |
| JACS | 5 | 10 | Oct 1922 | Data on Viscosity of Indiana Clay Slip with Electrolytes in Regard to the Casting of Terra Cotta | H.E. Davis | | | | | | | | | x | | | | | | | | | |
| JACS | 5 | 11 | Nov 1922 | Discussion on 'Notes on Shivering of Terra Cotta' | n/a | | | | | | x | | | | | | | | | | | | |
| JACS | 5 | 11 | Nov 1922 | The Effect of Some Fluxes on the Absorption and Transverse Strength of a Terra Cotta Body | E.C. Hill | | | | | | | | | x | | x | | | x | | | | |
| JACS | 6 | 1 | Jan 1923 | Looking Backward in the Terra Cotta Field | A.F. Hottinger | | | | | | x | | | | | | | | | | | | |
| JACS | 7 | 7 | July 1924 | Address on Terra Cotta | William H. Powell | | | | | | x | | | | | | | | | | | | |
| JACS | 7 | 7 | July 1924 | Clay Preparation | T.A. Klinefelter | | | | | | x | | | | | | | | | | | | x |
| JACS | 7 | 11 | Nov 1924 | Terra Cotta Body Investigation | B.S. Radcliffe | | | | | x | | | | | x | | x | | x | x | | | |
| JACS | 8 | 1 | Jan 1925 | A Study of Slips for Standard Finish Terra Cotta (Cones 4-5) | H.E. Davis and J.S. Lathrop | | | | | | | x | | | | | | x | | | x | | |
| JACS | 8 | 2 | Feb 1925 | Better Terra Cotta Slabs | F.B. Allen | | | | | | | | x | | | x | x | | | | | | |
| JACS | 8 | 2 | Feb 1925 | Notes on Terra Cotta Body Shrinkage | P.G. Larkin and E.R. Curry | | | | | | | | | | | | x | | | | | | |
| JACS | 8 | 9 | Sep 1925 | Symposium on Casting Problems | n/a | | | | | | x | | | | | | | | | | | | |
| JACS | 9 | 2 | Feb 1926 | Monograph and Bibliography on Terra Cotta | Hewitt Wilson | | | | | | x | | | | | | | | | | | | |
| JACS | 9 | 7 | July 1926 | Grog for Terra Cotta | D.F. Albery | | | | | | x | | | | | | | | | x | | | |
| JACS | 9 | 7 | July 1926 | An Attempt to Secure a Uniform Mixture of Fine and Course Particles in Grog from a Bin | W.A. Hull | | | | | | x | | x | | | | | | | | | | |
| JACS | 9 | 11 | Nov 1926 | Some Fundamentals of Terra Cotta | Harry Spurrier | | x | | | | | | x | | | | | | x | | | | |
| JACS | 9 | 11 | Nov 1926 | Shivering | W.J. Stephani | | | | | | | x | | | x | | | x | | | | | |

| Journal* | Volume | Issue | Date | Title | Author | Natural Weathering | Artificial: Freeze Thaw | Artificial: Autoclave | Artificial: Salt Bath | Artificial: Quenching | Discussion Only | Glaze/Slip Content | Grog Size | Clay Additives | Clay Type | Compressive Strength | Mix Ratios | Glaze Content: Clay | Absorption | Grog Material | Glaze Additives | Ink Staining | Clay Prep |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| JACS | 10 | 10 | Oct 1927 | Dry Grinding Clay to Eighty-Mesh | D.F. Albery | | | | | | x | | | | | | | | | | | | x |
| JACS | 11 | 2 | Feb 1928 | A Warpage Study of Terra Cotta Clays | R.M. Murphy | | | | | | | | | | x | | x | | | | | | |
| JACS | 11 | 5 | May 1928 | Methods for Testing Crazing of Glazes Caused by Increases in Size of Ceramic Bodies | H.G. Schurecht | x | | x | | | | | | | | | | | | | | | |
| JACS | 11 | 5 | May 1928 | Some Experiments on Terra Cotta Slips | E.C. Hill | | | | | | | x | | | | | | x | | | | x | |
| JACS | 12 | 7 | July 1929 | The Relation of a Modulus of Grain Size to the Mechanical Strength of Sagger Mixtures | Raymond E. Birch | | | | | | | | x | | | x | | | | | | | |
| JACS | 13 | 4 | Apr 1930 | The Causes and Prevention of Bulging in Hand-Molded Refractory Shapes | Gordon B. Richardson | | | | | | x | | | | | | | | | | | | |
| JACS | 13 | 6 | Jun 1930 | Method of Measuring Strains between Glazes and Ceramic Bodies | H.G. Schurecht and G.R. Pole | | | x | | | | | | | | | | | | | | | |
| JACS | 13 | 10 | Oct 1930 | The Packing of Particles | A.E.R. Westman and H.R. Hugill | | | | | | x | | x | | | | | | | | | | |

** TACS = Transactions of the American Ceramic Society*

*JACS = Journal of the American Ceramic Society*

*3ai. Glaze and/or slip content*

Ceramicists understood that a glaze and its clay body had to “fit” together. Thus, experiments were not exclusive to clay body mixes and preparation; the contents of glazes and slips were also studied. Although many tests of this sort were carried out, the results are difficult to compile due to the varying percentages of materials used. For example, Cornwall stone was useful in preventing crazing, but too much of it could increase crazing.[16] Similar discrepancies were observed with the uses of feldspar and flint- sometimes the studies would give amounts used, but sometimes they only referred to using “more” or “less,” making the actual recommended percentages difficult to determine.[17]

Other materials experimented with included calcium oxide, magnesium oxide, and barium oxide, which were all found to have negative effects on the glazed surface. Zinc oxide was found to be beneficial, while tin oxide had no noticeable effect.[18]

Clay content in glazes will be discussed separately in this chapter.

*3aii. Grog size*

Studies of the effect of various grog particle sizes yielded complex results; fine, medium, and coarse-sized grains were each found to have advantages and disadvantages. For example, coarse grog reduced clay body cracking, but could increase glaze fit issues, including glaze adhesion, and result in voids in the clay body. Many ceramicists came to the conclusion that grog should be carefully graded, combining certain percentages of sizes.[19] These percentages

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[16] Davis, H.E. and J.S. Lathrop. “A Study of Slips for Standard Finish Terra Cotta (Cones 4-5).” *Journal of the American Ceramic Society* 8.1 (1925).
Hill, E.C. “Some Experiments on Terra Cotta Slips.” *Journal of the American Ceramic Society* 11.5 (1928).

[17] Griffen, Henry R. “Shivering and Crazing.” *Transactions of the American Ceramic Society* 2 (1900).
Hill, E.C. “The Effect of Glaze Composition on the Crazing of Terra Cotta.” *Journal of the American Ceramic Society* 4.1 (1921).
Davis and Lathrop. “A Study of Slips for Standard Finish Terra Cotta (Cones 4-5).”

[18] Hill. “The Effect of Glaze Composition on the Crazing of Terra Cotta.”

[19] Clare, R.L and D.F. Albery. “The Effect of Variation of the Size of the Grog in Terra Cotta Bodies.” *Journal of the American Ceramic Society* 3.5 (1920). *Footnote continued on next page.*

were not always given, however, and articles also recommended studying the effects of grog material and source.

### *3aiii. Clay body additives*

Salts present within a clay body, whether the source was the clay, grog, or water, often resulted in efflorescence, or a white powder covering the surface of the ware, also known as scum. Many additives were used to counteract this phenomenon; the most widely used was barium carbonate. It was said that the barium neutralized the salts, preventing the surface buildup that could often lead to spall.[20] Ceramicists also experimented with other additives, including barium hydrate, feldspar, and sodium carbonate. Barium carbonate was the most widely used in the industry, however, and it is still used today.[21]

### *3aiv. Clay type*

The impact of the type of clay used was often studied; of primary concern was the amount of sand in a clay. One study showed that sandy clays led to cracking in the clay body, but another found that they reduced firing cracks.[22] These contrasting results are inconclusive. Clays were also examined for their impact on shrinkage and warpage, both during drying and firing, and

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Allen, F.B. “Better Terra Cotta Slabs.” *Journal of the American Ceramic Society* 8.2 (1925).
Birch, Raymond E. “The Relation of a Modulus of Grain Size to the Mechanical Strength of Sagger Mixtures.” *Journal of the American Ceramic Society* 12.7 (1929).
[20] Gates, M.E. “A Satisfactory Method of Using Barium Hydrate in Terra Cotta Bodies.” *Journal of the American Ceramic Society* 3.4 (1920).
Carruthers, John L. “Notes on Shivering of Terra Cotta.” *Journal of the American Ceramic Society* 5.8 (1922).
Parmelee, Cullen W. “Soluble Salts and Clay Wares.” *Journal of the American Ceramic Society* 5.8 (1922).
Hottinger, A.F. “Looking Backward in the Terra Cotta Field.” *Journal of the American Ceramic Society* 5.11 (1922).
Wilson, Hewitt. “Monograph and Bibliography on Terra Cotta.” *Journal of the American Ceramic Society* 9.2 (1926).
[21] Patwardhan, Nirmala. *New Handbook for Potters*. New Delhi: Allied, 2005, 318.
[22] Hill, E.C. “Some Experiments on the Firecracking of Terra Cotta.” *Journal of the American Ceramic Society* 5.6 (1922).
Murphy, R.M. “A Warpage Study of Terra Cotta Clays.” *Journal of the American Ceramic Society* 11.2 (1928).

their tendency to produce shivering. Specific results for tests can be found in the article chronology which follows in Chapter *3b*, but the trends were scattered and contradictory.

*3av. Compressive strength*

It was understood that high compressive strength was important in a terra-cotta unit; a strong piece would be durable and able to resist building loads and weathering over the course of its lifetime. Tests were often carried out with the hope of improving on common practices in the industry to increase strength, but specific results of strength testing were not always given. One interesting test, however, consisted of measuring the compressive strength of samples before and after freeze-thaw testing.[23] It was noted that the cyclic testing reduced the strength of the terra cotta.

*3aii. Clay body mix ratios*

The proportion of clay to grog was sometimes discussed and tested. 3:1 was a common ratio, but one tester considered 3:2 normal.[24] A ratio of 1:1 was used in one test, in which two types of clay were used (vitrifying and open-burning).[25] A 4:1 ratio was used in a test examining slab mixtures.[26] Clearly, there was much variety in the industry, but it was usually common for the clay percentage to outweigh that of grog.

*3avii. Glaze composition: clay content*

In addition to the previously discussed additives in glazes, the clay content of glaze mixes was often discussed. Less than 50% clay content yielded glazes with greater fusibility and increased vitrification, which in turn decreased crazing and other glaze defects.[27] Too little clay content,

[23] Minton, R. H. "The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies." *Journal of the American Ceramic Society* 1.3 (1918).
[24] Radcliffe, B.S. “Terra Cotta Body Investigation.” *Journal of the American Ceramic Society* 7.11 (1924).
[25] Birch. “The Relation of a Modulus of Grain Size to the Mechanical Strength of Sagger Mixtures.”
[26] Allen. “Better Terra Cotta Slabs.”
[27] Hill. “Some Experiments on Terra Cotta Slips.”

however, could lead to unfused glazes.[28] Generally, anywhere from 20-50% clay content was recommended. Altering clay content also altered the color of a glaze, though, and thus this strategy was not used widely with polychrome glazes.

*3aviii. Absorption*

Ceramicists often discussed the absorption rate of terra cotta; it was a given that lower rates were ultimately beneficial for the durability of a ware. One interesting test measured the rates at which water flowed into units before and after freezing and thawing.[29] It was found that this cycle increased the rate at which water flowed into a unit, and thus it was concluded that freeze-thaw cycles were detrimental to terra-cotta units.

*3aix. Grog materiality*

Some experiments undertaken examined the materials used for grog. The type of material and its source were usually wide-spread, and this made its properties hard to control and predict. One author advocated that a material be manufactured specifically to be used as terra cotta grog in order to control quality.[30] Another test examined the use of furnace slag as grog, and found the results to be beneficial. The terra cotta produced was durable, light-weight, and cheaper.[31] However, no further mention of this grog material was ever found by this author.

*3ax. Glaze additives*

Ceramicists sometimes experimented with additives in glaze mixtures. These materials ranged from tin oxide and magnesium carbonate to feldspar and Cornwall stone. The results were scattered, however, and not numerous enough to draw conclusions.

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[28] Stephani, W.J. “Shivering.” *Journal of the American Ceramic Society* 9.11 (1926).
[29] Spurrier, Harry. “Some Fundamentals of Terra Cotta.” *Journal of the American Ceramic Society* 9.11 (1926).
[30] Albery, D.F. “Grog for Terra Cotta.” *Journal of the American Ceramic Society* 9.7 (1926).
[31] Minton. "The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies."

*3axi. Ink staining*

Authors occasionally examined a glaze surface for cracks or crazing by allowing ink or dirty coal water to sit on the surface.[32] The liquid would then by wiped away, and hairline cracks would be more visible. This technique demonstrates that ceramicists understood that cracks could be present even if they weren't visible to the naked eye, and that these imperfections were worth noting.

*3axii. Clay preparation*

Although never directly associated with testing, some authors recommended proper preparation of clay before mixing the clay body to be fired.[33] Processes recommended included washing, screening, filtering, and grinding the clay to remove impurities.

*3axiii. Weathering*

Although these tests were driven by a desire to make terra cotta a more durable building material, the longevity of the design mixes was rarely tested in a conclusive manner. Some experiments exposed their units to natural weathering, letting them sit outside in a yard for anywhere from six months to twelve years. Shorter amounts of time usually did not yield crazing, but twelve years sometimes did.[34] Although this line of thinking demonstrated a step towards understanding the impact of weathering on terra cotta, the methods used were not always accurate predictors.

Occasionally, more advanced techniques were used to predict glaze failure. Today these procedures are referred to as artificial or accelerated weathering; historic literature showed four

[32] Wilson, Hewitt. "Notes on Terra Cotta Slips with Reference to the Use of Asbestos and Chlorite Mica." *Journal of the American Ceramic Society* 3.2 (1920).
Hill. "Some Experiments on Terra Cotta Slips."
[33] Albery, D.F. "Dry Grinding Clay to Eighty-Mesh." *Journal of the American Ceramic Society* 10.10 (1927).
Klinefelter, T.A. "Clay Preparation." *Journal of the American Ceramic Society* 7.7 (1924).
[34] Schurecht, H.G. "Methods for Testing Crazing of Glazes Caused by Increases in Size of Ceramic Bodies." *Journal of the American Ceramic Society* 11.5 (1928).

techniques. One was a freeze-thaw test, very similar to modern techniques.[35] The samples were saturated, frozen, and then thawed. This technique never produced rupturing or dramatic failure, but it did measurably weaken the bodies subjected to the tests in terms of absorption and compressive strength. An extreme version of this test, called “quenching” was once carried out; it involved heating a piece to a high temperature and then dropping it in ice water.[36] This would usually quickly show results of cracking.

Since it was understood that salts were detrimental to terra cotta in many ways, a sodium sulfate boil test was carried out.[37] Pieces subjected to testing were boiled in the solution and then dried at room temperature; repeated cycling yielded slight flaking. Steam pressure was also sometimes applied using an autoclave.[38] This was done to simulate expansion due to moisture, and sometimes resulted in crazing.

Although these methods of artificial weathering were not always effective in producing glaze or body failure, they show that the ceramicists conducting tests had an idea about what it meant for a ware to be truly durable over time.

[35] Minton. "The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies."
Spurrier. “Some Fundamentals of Terra Cotta.”
[36] Radcliffe. “Terra Cotta Body Investigation.”
[37] Minton. "The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies."
[38] Schurecht. “Methods for Testing Crazing of Glazes Caused by Increases in Size of Ceramic Bodies.”
Schurecht and Pole. “Method of Measuring Strains between Glazes and Ceramic Bodies.”

**3b. List of Tests- Chronological**

February 1900: Edward Orton, Jr. “Note on the Relation between the Tensile Strength of Clay Mixtures and the Size of the Grains of their Non-Plastic Constituents.” *Transactions of the American Ceramic Society*.

The author discussed the effect of grain size on clay plasticity. In order to better understand these properties, sands of six various carefully-graded sizes were blended with a plastic ball-clay into brickettes to test for tensile strength. The pieces were dried (but not fired) and the weight of water required to snap the discs measured with a homemade mechanism. It was found that the tensile strength of a brickette related inversely to the diameter of the sand grains- within limits. It was worried that too fast drying may have caused shrinkage cracks in some of the fine-sand pieces, skewing the results.

February 1900: Henry R. Griffen. “Shivering and Crazing.” *Transactions of the American Ceramic Society*.

The author described his personal experiences in glaze defects. He noted that thinner glazes resulted in crazing, and thicker glazes resulted in shivering; these observations were made in 1877. However, when this glaze was applied to a different body, the results were the opposite- crazing on thick glaze, and no defects on thin glaze. It was understood that this was due to differences in the coefficients of expansion between the glaze and clay body. The addition of flint or feldspar to the clay body was found to be an effective means of adjusting these values. However, the combinations of materials added to different types of clay bodies and glazes at different temperatures were often contradictory, and made it difficult to discern factual explanations.

February 1902: William H. Zimmer. “Crystallized Glazes.” *Transactions of the American Ceramic Society*.

The author argued that in order to better understand ceramic bodies, his colleagues should learn to study their wares under the microscope. He proclaimed that using test tubes and a balance was not enough, and microscopes would allow for further areas of study. He went on to describe the nature of crystallized glazes that were primarily used on pottery.

February 1902: Ross C. Purdy. “Stoneware Glazes.” *Transactions of the American Ceramic Society*.

The author began by defining stoneware as “a grade of pottery made from a natural clay or mixture of clays, which is burnt to vitrification, and in which the body and glaze are matured in one and the same fire. The articles usually made were crude jugs, crocks, receptacles for fruits and dairy products.” Although terra cotta was not specifically discussed, the discussion of glazes is still relevant for this thesis, as it described the methods being explored and ceramicists’ ways of thinking. The glazes discussed are as follows:

- Salt glazes: Used less frequently, this glaze methodology involved coating with a slip or Bristol glaze, and then exposing the piece to salt vapors during firing.
- Slip glazes: These glazes were intended to fuse and vitrify with the body, and were made of “ferruginous” clays, usually resulting in a red, brown, or black color.
- White Bristol glazes: Being a new material, its properties had not been studied much by chemists. The oxygen ratio and $Al_2O_3$ ratio were examined; fusibility could be controlled, but shrinkage and other “physical properties” were beyond chemical calculation. Too much clay content was found to result in crawling. Increased $K_2O$ and $CaO$ increased the glaze’s hardness. Zinc was often attributed to crawling, but the author did not find this to be true. Other specific chemical results were discussed.

February 1902: Stanley G. Burt. “Note on Fritting.” *Transactions of the American Ceramic Society*.

Fritting a glaze, or pre-fusing all of the fluxes to be used, aided in permanently contracting the glaze, which was thought to help prevent crazing. It was understood that a clay body expands and contracts with changes in moisture content and temperature, and that this was often a source of crazing. It was unclear how exactly the impact of a fritted glaze affected this phenomenon. The author noted that boracic acid was often used to counteract crazing, but did not go into detail about this procedure. He concluded by stating that there was still much to be understood, and it was the Society’s responsibility to study and explain the facts.

February 1902: Edward C. Stover. “Bacterial Growth as a Factor in Ageing Clay Mixtures.” *Transactions of the American Ceramic Society*.

The author discussed the long tradition of ageing clay body mixtures, which prevented drying and fire cracks and increased plasticity. He described this as a process of the mixture truly becoming a compound, not merely separate materials mixed together. After conducting experiments, the procedures of which were not specified, he concluded that bacterial growth aided in this process. He believed this was due to the sulphureted hydrogen gas given off by the bacterial species.

February 1902: Lawrence E. Barringer. “The Relation between the Constitution of a Clay, and its Ability to Take a Good Salt Glaze.” *Transactions of the American Ceramic Society*.

The author cited that not much research had been done on the process of salt glazing. Chips of salt glaze which had adhered well to bricks were ground and analyzed, and the chemical content was compared to that of the bricks. Of particular interest was the amount of lime present: the clay had 0.725%, while the glaze had 3.5%. The discrepancy was attributed to efflorescence; it was thought that the soluble salts in the body created deposits on the surface, which were absorbed into the glaze. Tests were also carried out which indicated that too high of an alumina-silica ratio in the clay would prevent a good glaze from adhering and creating a uniform surface; a 1:10 molecular ratio was recommended. It was also noticed that finer sand resulted in glazes of lighter colors.

February 1902: Francis W. Walker. "The Fluxing Power of Mica in Ceramic Bodies." *Transactions of the American Ceramic Society*.

The author found, after conducting experiments using clay bodies made with materials in graded proportions, that mica, when ground finely, worked as a flux, but was not as effective as the commonly used feldspar.

February 1902: Ray Thomas Stull. "Notes on the Development of Greens from Cupric Oxide in Glazes." *Transactions of the American Ceramic Society*.

The author sought to create a formula for a green glaze without the use of copper, since copper was volatile and tinted saggers. A large number of glazes were made using combinations of $CuO$, $CaO$, $K_2O$, $Na_2O$, $PbO$, $BaO$, $ZnO$, $MgO$, $Al_2O_3$, $SiO_2$, and $B_2O_3$. Results were detailed for all combinations tested, discussing colors produced and recommended proportions of additives. Black and white photographs are included of all samples; some show signs of crazing or crawling. The author stated that these could have been corrected by changing the mixing process, which was not done for this experiment in order to obtain consistent procedures between samples.

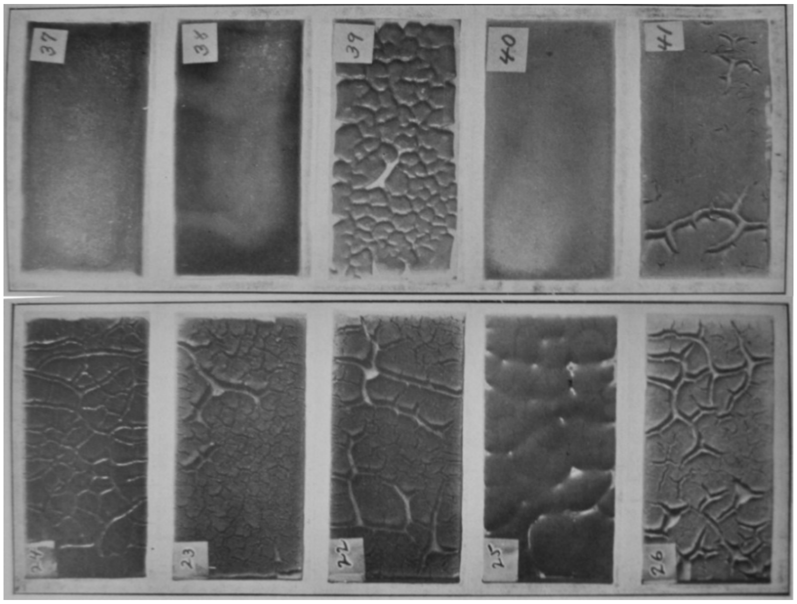


Figure 10: Some samples of green glazes. Source: *Transactions of the American Ceramic Society*, v4.

<u>February 1903</u>: Willard D. Richardson. “The Work of the Ceramic Engineer in the Brick-Making Industry.” *Transactions of the American Ceramic Society*.

Engineering was, at this time, growing rapidly as a profession and becoming more commercially viable. The author noted that, in particular, ceramic engineering departments were beginning to be formed at some universities. Although these engineers primarily focused on pottery and artistic applications, the author advocated for the inclusion of “not only all forms of building brick, paving brick and fire brick, but also drain tile, sewer pipe, roofing tile and terra cotta.” He then went on to describe financial and logistical challenges of the brick industry, and the aid these engineers could supply.

<u>March 1918</u>: R.H. Minton. "The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies." *Journal of the American Ceramic Society*.

The article noted that dunting, or sudden rupture, was a common failure type. Previously accepted knowledge stated that "the first and most important cause of failure is a defective body mixture." Ceramicists also understood that the rate of firing and cooling also played a role in the tendency of a terra-cotta unit to crack. It was also noted that sandy clays were more likely to yield cracking than more fatty, plastic clays.

The author went on to comment that although grog size and quantity had been studied, the material used had been overlooked in the industry. He proposed that furnace slag be used because it was cheap, light-weight, and possessed "sufficient resistance to destruction by the elements." Granulated slag, rather than crushed slag, performed better, because it was lighter and contained no iron. Experiments were carried out to evaluate the performance of furnace slag as grog. Bodies were prepared following “normal” terra cotta-making procedures, and two types were made- one with slag, and one with normal grog. After being fired at cone 5 for 45 hours and cooled, the samples were tested for absorption rates, and then allowed to weather naturally for one year. At the end of this year, no shivering, crazing, or other defects could be seen, and thus "so-called artificial freezing tests" were carried out.

Samples were boiled for three hours in a sodium sulphate solution, and then dried at room temperature for 48 hours. This cycle was repeated with the hope of causing visually evident damage. After 15 repetitions, the body prepared with grog showed signs of flaking, but the slag body resisted 34 cycles.

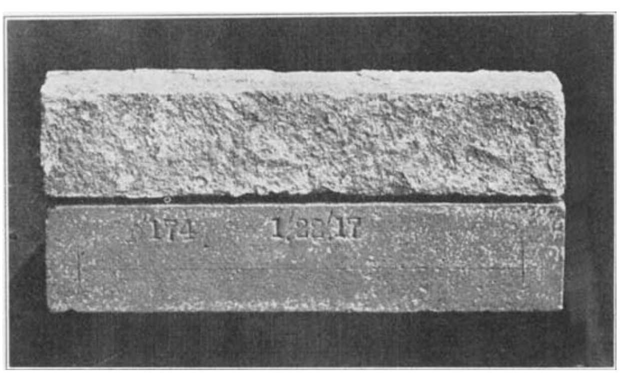


Figure 11: Grog (above) and slag (below) bodies after boiling cycles. Source: *Journal of the American Ceramic Society*

Tests were then conducted to expose the samples to "actual freezing," as they were saturated with water, frozen at 5°F, and thawed. Although 25 cycles did not visually affect the samples, decreased compressive strength was measured- the body made with normal grog lost a greater percentage of resistance. Although wear and tear could be seen, dunting never occurred in either sample.

**Note:** This article is later used as a guide for the historic boiling salt bath test.

**Note:** This article is similar, but less specific, to the procedures used for a contemporary freeze-thaw test.

<u>May 1918</u>: Hewitt Wilson. "Polychrome Decoration of Terra Cotta with Soluble Metallic Salts." *Journal of the American Ceramic Society*.

This article noted that when glaze was painted onto the clay body, cracks were often developed in the glaze, which was attributed to the unevenness of brushstrokes. Another technique often employed was spraying the glaze through "masques" (stencils). Although this left uneven lines between colors, it was a good technique for large pieces of terra cotta. Overglaze spraying involved spraying layers of varying colors and was recommended for small pieces with complicated designs.

The new method being proposed was painting with soluble metallic salts. The salts were dissolved in glycerine and painted onto dry pieces of terra cotta, dried at 120°C to set the glycerine, then fired. The glaze was then applied, at which point a second firing took place.

The article discussed formulas of the various colors employed and the results of color tests.

September 1918: Cullen W. Parmelee. "An Unusual Cause of Spalling of Sewer Pipe." *Journal of the American Ceramic Society*.

The source of spall was investigated in this article when damage could be seen during the burning (firing) stage of sewer pipes. The surface of the pipes appeared to be flaking, and the author was brought in to discover the cause. It was found that roughly spherical pieces of unknown makeup were embedded in all of the spalled flakes. Closer inspection found these spheroids to be geodes, ranging in diameter from two to three millimeters. The geodes were discovered to have come from sandstone in the clay quarry, and although the larger six-inch pieces were filtered out, the smaller ones were fired in the terra cotta, causing the damage to the body.

October 1918: G.H. Brown. “Notes on Sagger Clays and Mixtures.” *Journal of the American Ceramic Society*.

The durability of sagger clay mixes was described in this article. Lack of durability had been an issue in the industry, and failure could be characterized in the following ways: breakage due to rough handling or rapid heating, breakage during cooling, or deformation of loaded saggers at the maximum kiln temperature. It was noted that sometimes cheaper clays produced saggers with longer lives. Desirable properties of saggers were listed as the following: refractoriness (high melting point), mechanical strength, resistance to deformation, and resistance to changes in temperature. The author divided saggers into two categories: vitrifying, which vitrified at low temperatures, had low porosity, and had high but uniform shrinkage, and open-burning, which was porous, with a high absorption rate at high temperatures, and had low shrinkage. A good proportion was cited as being 50% grog, 20% vitrifying clay, and 30% open-burning clay. He also noted, however, that it was not enough to classify the sagger as vitrifying or open-burning; the composition of the raw materials must also be understood.

May 1919: “Professional Divisions.” *Journal of the American Ceramic Society.*

The Terra Cotta Division of the American Ceramic Society had been fully organized, and work to be done was planned out. The Division was comprised of 49 members from 24 companies, the Bureau of Standards, and Ohio State University. Investigations that the article cited as being carried out include “the improvement and development of glazes; the effect of electrolytes on terra-cotta bodies; the effect of the size of grog in terra-cotta bodies; spalling of terra-cotta bodies; spraying machines; humidity drying; firing terra cotta kilns; standardization of terra cotta colors; etc.” The results had yet to be prepared and submitted at the time of publication.

January 1920: E.C. Hill. “Some Data on the Development of Terra Cotta Glazes.” *Journal of the American Ceramic Society.*

The article described the study of Bristol glazes (those containing feldspar, flint, clay, whiting, and zinc oxide). Experiments were undertaken to understand the impact of tin oxide, barium and magnesium carbonates (commonly used), and strontium carbonate (not commonly used) when added to this type of glaze. It was found that a clay content of 0.20 to 0.25 yielded more fusibility of glazes than 0.05, but the presence of CaO reversed this. Other additives were experimented with, but without conclusive results.

February 1920: Hewitt Wilson. “Notes on Terra cotta Slips with Reference to the Use of Asbestos and Chlorite Mica.” *Journal of the American Ceramic Society.*

The articles focused on white or light gray vitrified slip coatings, fired at cone 6. When vitrified slip was used as an underslip (i.e. covered with glaze), there was often poor adhesion to the clay body noticed, resulting in peeling. Also, if the slip itself was applied too thickly, it was likely to crack. Cracks could also be caused by large grog in the slip- this was believed to be caused by a “roughening” of the surface, due to the large particles.

Before the underslip was applied, the clay body could be finished in two ways- either with a steel tool, such as a trowel, or with a water brush (similar to sponging) when the body had reached a leather-hard state. After firing, units were examined by the following methods: visual comparison of color and surface irregularities; absorption was tested by painting on red ink and washing away after 10 minutes; cracking was examined by observation after washing with "a slip of coal dirt and water" to bring out invisible cracks; crazing could be seen after these ink and dirty water treatments. It was noted that a unit that looks to be free of cracks and crazing to the naked eye may still in fact have imperfections that are more difficult to see.

The author concluded with the following:

> The physical condition of the body upon which vitrified slips and glazes are sprayed is of fundamental importance in the production of perfect surfaces. The maximum size of grog grains and the proportioning of the grog sizes, the methods of pressing, finishing, and spraying are all of utmost importance. While the composition of the slip needs thorough investigation, we would suggest that the shape of the individual particles be also investigated for the production of unbroken coatings. This includes the study of the magnesia and magnesia-alumina silicates as represented by asbestos, steatite, and the minerals of the general mica division.

<u>April 1920</u>: M.E. Gates. "A Satisfactory Method of Using Barium Hydrate in Terra Cotta Bodies." *Journal of the American Ceramic Society*.

The article noted that barium carbonate was often used to "take care of," or neutralize, soluble salts in terra-cotta clay bodies to prevent spall and efflorescence. When barium hydrate was used, if it came in contact with the surface of the unit, the presence of air would cause it to change to barium carbonate and leave a "white scum" on the surface, which appeared to be harmless. The author then went on to describe his designed method for keeping the barium hydrate in solution before its addition to the clay body mix, involving various mixing tanks. Because barium hydrate was more soluble than barium carbonate, it came in contact with more salts, and thus made it more effective and economical.

May 1920: R.L. Clare and D.F. Albery. “The Effect of Variation of the Size of the Grog in Terra Cotta Bodies.” *Journal of the American Ceramic Society*.

The quality of grog, including composition and hardness, was hard to regulate, and thus the size and amounts were experimented with. The author detailed a study, in which three types of body mixes were created, each composed of one-third grog and two-thirds clay; grogs of coarse, medium, and fine sizes were employed (tables with the grading of all grog sizes were included). Half of the samples were covered in a glaze, half in a vitreous slip- firing times and temperatures were also varied. After firing, all pieces were naturally weathered for six months to allow for the development of cracks and crazing. The fine-grog body yielded a tight sound, more warping, more cracking, less absorption, medium shrinkage, and good glaze coverage. Conversely, the coarse-grog body yielded a shaky sound, no warping, no cracking, greater absorption, little shrinkage, and poor glaze coverage. The author ultimately concluded that controlled grog quality would be recommended.

June 1920: F.B. Ortman. “The Composition of Kiln Gasses and Their Effect on Terra Cotta Glazes and Colors.” *Journal of the American Ceramic Society*.

Experiments were carried out to verify the presence and determine the source of sulphur, which could result in bubbling, pin-holing, or other glaze defects. Possible sources postulated were the clay, grog, and coal used to fire the kiln, and coal was then verified as the source. Experiments were then carried out with variations in kiln gasses and ventilation strategies. Ultimately, the author hoped to create a standard for kiln ventilation, which would create more stable glazes.

January 1921: E.C. Hill. “The Effect of Glaze Composition on the Crazing of Terra Cotta.” *Journal of the American Ceramic Society*.

The author began by noting that crazing was primarily caused by the compositions of the body, engobe, and glaze used. The chance of crazing was also increased by rapid cooling, immature firing, and heavy coats of glaze and/or engobe. Specific to body composition, it was noted that fat, plastic clays were more likely to produce crazing than sandy ones, but very sandy clays could produce shivering. The author said that because there were too many varieties of clay to consider, it would be most effective to focus on engobes and glazes in the prevention of crazing.

After carrying out experiments, it was found that ZnO was most effective at preventing crazing, and feldspar was also effective. MgO, BaO, and CaO, however, tended to encourage crazing. $SnO_2$ and flint had no effect. It was also found that an increased amount of clay in the glaze also helped to prevent crazing, and required the addition of less material.

June 1921: R.L. Clare and R.N. Long. “The Value of Ageing the Terra Cotta Body.” *Journal of the American Ceramic Society*.

The article discussed improvements made by ageing clay before shaping the terra-cotta unit. An experiment was carried out in which the clay was aged twelve days, which increased plasticity and workability. It was also found that the mixture resulted in a straighter and stronger dried piece. The author ultimately concluded by recommending this process for the terra-cotta industry.

June 1922: E.C. Hill. “Some Experiments on the Firecracking of Terra Cotta.” *Journal of the American Ceramic Society*.

Firecracking was defined as “sharp, hair-line cracks extending into the body” of terra cotta-these were also referred to as cooling cracks or dunting. Units tended to crack after weathering, but rapid cooling was also a frequent cause. The author stated that “the rate of cooling has a much greater effect on the tendency to firecrack than the composition or physical properties of the body.”

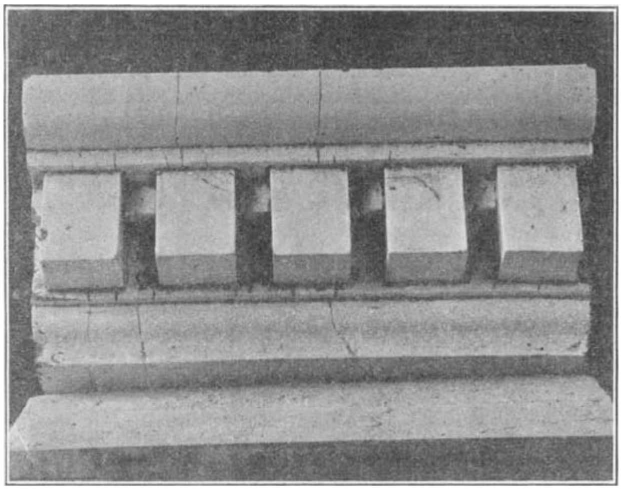


Figure 12: Typical firecracking. Source: *Journal of the American Ceramic Society*

In the experiments carried out, units were made to test for firecracking, linear fire shrinkage, transverse strength, and absorption. Specific results were given for each of six clays that were employed in the study.

Firecracking was likely to occur when the clay body was composed of a sandy clay, due to the difference in expansion and contraction between the sand and clay. Coarse grog yielded less firecracking, and it was noted that size and quantity were more important than the type or source of grog. It was also noted that cracks could develop over time, and might not be immediately apparent. Pieces in projecting parts of a building were also found to be more likely to crack, due to their increased exposure to sun heating and cooling at night. Strains that began during kiln cooling were noted to be worsened by weathering. The pieces in this experiment were naturally weathered for three years, and yet did not reveal any cracks.

The author noted that the cooling needs of a terra-cotta unit varied by the size of a piece. Firing units of varying sizes together was ill-advised, as cooling times and rates became difficult to calculate.

It was also found that the tendency of a piece to crack was not dependent on its rates of absorption, porosity, or the strength of the clay- sandy material was a more influential factor.

However, units of low porosity and high strength were recommended to counteract spall caused by freeze-thaw cycling. The author noted that although this went against convention of the time, his experiments proved otherwise, as long as the proper clay was used.

August 1922: John L. Carruthers. “Notes on Shivering of Terra Cotta.” *Journal of the American Ceramic Society*.

In order to determine methods of avoiding shivering on terra-cotta units, the author carried out a series of experiments, with the understanding that the body and glaze must “fit.” Previous investigations had found that shivering could be caused by siliceous clay or soluble salts, and that feldspar or high clay contents prevented shivering. After carrying out trials using units made from various types of clay, with different amounts and types of grog and sand, the author concluded the following: shivering may be caused or promoted by clay with high silica content, finely ground siliceous grog, or soluble salts (in the clay, grog, or tempering water). It was also found that shivering could be avoided by adding feldspar or felsite as a flux to increase density, using coarse sand or grog (to open up the body), or adding barium carbonate to counteract the soluble salts.

August 1922: Cullen W. Parmelee. “Soluble Salts and Clay Wares.” *Journal of the American Ceramic Society*.

The article defined efflorescence as surface deposits due to soluble salts in clay, and scum as salt due to “action of gases upon the wares during drying or burning,” but also noted that the terms were often used interchangeably. Barium was often added to terra-cotta clay bodies to prevent salt deposits. It was found that adding common salt and soda ash, or sodium carbonate, produced salt upon drying, but there was no salt left on the burned ware.

October 1922: “Discussion on ‘Some Experiments on the Fire Cracking of Terra Cotta.’” *Journal of the American Ceramic Society*.

This is a conversational dialogue, discussing the importance of the June 1922 article. The work was well-received, and contributors agreed with the author’s findings, praising its relevance.

October 1922: C.W. Hill. “Terra Cotta Problems Suggested for Discussion and Investigation” *Journal of the American Ceramic Society*.

This article did not chronicle any testing done by the author, but rather compiled proposed ideas and theories to be further studied. The list was comprised of plaster of Paris, body mix and materials (including barytes, grog, and flux), glaze, processes (including patching), and service problems. Of particular interest were the ideas that underslip might “lower glaze adherence” to the clay body, and the effect of grog sizes on slip cracking.

October 1922: H.E. Davis. “Data on Viscosity of Indiana Clay Slip with Electrolytes in Regard to the Casting of Terra Cotta” *Journal of the American Ceramic Society*.

The article focused on one specific clay, Indiana clay, which was commonly used in the Chicago area. Tests were carried out adding sodium carbonate, sodium silicate, combinations of the two, gallic acid, and tannic acid to determine the viscosities of the clays and their applicability for casting units. Sodium carbonate, used alone, was found to be most effective. Tannic acid was also helpful, but the vast quantity required made it impractical for commercial applications.

November 1922: “Discussion on 'Notes on Shivering of Terra Cotta.'” *Journal of the American Ceramic Society*.

This is a conversational dialogue, discussing the merits of the August 1922 article. The article was well-received, with additional comments made. It was noted that crazing may increase with weathering of terra cotta, but shivering did not advance over time. Also, more finely ground silica was noticed to result in less shivering. The idea of a body and glaze “fitting” together was brought up again.

<u>November 1922</u>: E.C. Hill. “The Effect of Some Fluxes on the Absorption and Transverse Strength of a Terra Cotta Body.” *Journal of the American Ceramic Society*.

This article chronicled experiments that the author carried out in an attempt to lower absorption and raise the strength of terra-cotta bodies. Alkali salts were found to be the most effective fluxes, but not productive in practice due to the resulting scum left on the surface of the units. Insoluble materials high in alkali were ultimately the most desirable. Examples of these that were tested include feldspar, powdered glass, and cryolite. The author also noted that the amount of advantage gained by using a flux also depended upon the nature of clay and grogs used in the body mix.

A conversational dialogue followed the main text of the article, contributed to by multiple professionals. It was noted that the content and size of glass powder should also be considered, and that a constant composition would be important. One contributor noted that a low-burning clay might also make an effective flux. He suggested that an “artificial flux,” such as those mentioned in the article, may have a negative effect on the terra cotta’s warpage, and that the amount of grog used would have to be proportionally altered.

<u>January 1923</u>: A.F. Hottinger. “Looking Backward in the Terra Cotta Field.” *Journal of the American Ceramic Society*.

This article gave a short history of the terra-cotta industry and some techniques employed. It noted that terra cotta was not used structurally until the introduction of buff burning clays (as opposed to the earlier red burning clays) and grog to control shrinkage. The author claimed that “it is notable that very few cases of failure of terra cotta through weathering or freezing exist,” which he attributed to the hard-burned nature of the material. Barium carbonate, used to prevent sulphates from creating efflorescence on the surface, was introduced in 1885. Ceramic chemists were involved at this time, controlling burning times and temperatures to control oxidation.

The author claimed that glazed or enameled terra cotta was introduced to grant an easily cleaned and light-reflecting material for the new skyscrapers. It then grew in popularity, first being produced in a two-fire process, but single-fire processes were later developed, and in use at the time this article was written. The article concluded hopeful for further future developments of the industry, despite the large amount of work it would require.

<u>July 1924</u>: William H. Powell. “Address on Terra Cotta.” *Journal of the American Ceramic Society*.

This article was not based on factual testing, but rather read more as a narrative. The author claimed the need for a consistent use of nomenclature in the industry. It was also mentioned that the development of a patching material (either for factory or on-site usage) would be beneficial. The types of failure or their causes that resulted in the need for this type of patching were not given.

<u>July 1924</u>: T.A. Klinefelter. “Clay Preparation.” *Journal of the American Ceramic Society*.

This article discussed the merits of washing, screening, and filtering clay before mixing it into the terra-cotta body. This process removed impurities, which could cause discoloration or “pop outs” in the fired ware. The author acknowledged that he did not understand the science behind these “pop outs,” but observed improvements when certain procedures were followed. He continued by suggesting, in detail, the process to be followed: washing the clay, passing it through 80-mesh, and then “filter pressing.” After that point, it could be mixed with the necessary grog. The author also stressed the importance of utilizing clean grog, free of impurities. It was understood that bits of brass would result in “the old familiar friend, ‘Mr. Green

Spot.'" The author acknowledged that this process as a whole was expensive and not always possible, but recommended it especially for lower story pieces, where ornament was more visible.

November 1924: B.S. Radcliffe. "Terra Cotta Body Investigation." *Journal of the American Ceramic Society*.

Experiments were conducted to test varieties of clay and grog types and test for properties of terra-cotta units. Samples were made using five types of clay and two types of grog- a chemical analysis was included for all five clays. The first grog used was the authors "regular" grog, and the second was made by calcining each of the clays. Each body was made using a 60:40 clay:grog ratio, and an additional control batch was made with only clay. Pieces were made to test for shrinkage, absorption, and modulus of rupture (sizes and shapes of the units varied by test). After the pieces were fired, all tests were carried out. Additionally, a quenching test was done, which involved heating a piece to 400°C for one hour, and then dropping it in ice water. This was repeated until the unit cracked, usually by three trials.

Detailed tables with data from all tests were given. Clays were ranked by their behavior in each test (for example: Goose Lake clay [one of the local types of clay tested] was the most resistant to cool-cracking). The cool-cracking and modulus of rupture tests were found to be related. It was also noted that $Al_2O_3$ and $SiO_2$ content in the clay was important for successful tests.

The article was followed by a discussion section, in which audience members asked clarifying questions about the tests.

January 1925: H.E. Davis and J.S. Lathrop. “A Study of Slips for Standard Finish Terra Cotta (Cones 4-5).” *Journal of the American Ceramic Society*.

The authors sought to create a slip that would be vitreous, white, have a wide firing range, and be free of crazing, checking, peeling, and flaking; and to understand the limits of Cornwall stone, feldspar, flint, and clays in terra-cotta slips. After firing various samples made of different proportions of materials, they found that high clay content yielded crazing and decreased vitrification, more than 50% Cornwall stone also produced crazing, 35% feldspar was beneficial, flint produced crazing, whiting decreased the firing range, and finally that adding soda ash had no impact as a flux. No combination of materials experimented with produced a vitreous slip that was also white, and crow’s-footing was never entirely eliminated. Their ultimate recommendations were as follows:

- Cornwall stone: 25-50%
- Feldspar: 0-35%
- Clay: 25-55%
- Flint: 0-10% (or less)

February 1925: F.B. Allen. “Better Terra Cotta Slabs.” *Journal of the American Ceramic Society*.

The author listed the following as desirable properties for terra-cotta slabs:

- High refractoriness: resistance to warpage or deformation under load
- Mechanical strength
- Accurate dimensions
- Resistance to sudden temperature changes
- Resistance to spall (or “spit-out”)
- High thermal conductivity, yielding even heat distribution

- Resistance to abrasion

Although historically, slabs were mixed with 26% grog, 50% low plasticity clay, and 24% bonding clay, the author sought to improve this mixture. Grog content was increased to 35%, with greater variety in size. This yielded harder slabs, but was “too refractory.” Next, 30% grog was used, and the results were satisfactory, exuding the previously listed requirements. He also recommended increasing the terra cotta clay percentage and slightly decreasing those of the non-plastic clay and the grog.

<u>February 1925</u>: P.G. Larkin and E.R. Curry. “Notes on Terra Cotta Body Shrinkage.” *Journal of the American Ceramic Society*.

The article chronicled the author’s quest to remedy the process of shrinkage control in terra cotta. The proposed method involved crushing all materials (clay, grog, etc.) separately and then combining them to form the clay body mix. The author noted that previously, clay was not crushed, resulting in a lumpy texture. He also recommended increasing the terra-cotta clay percentage and slightly decreasing those of the non-plastic clay and the grog.

The amount of water content and respective shrinkage in samples was also discussed: water content varied from 25.2-29.0% and shrinkage from 6.07-7.04%.

<u>September 1925</u>: “Symposium on Casting Problems.” *Journal of the American Ceramic Society*.

This article is the transcript of a conversational symposium, held to discuss issues that arose with attempts to cast terra cotta. Questions asked included whether a higher firing temperature or longer firing period (referred to as “soaking” when held at a constant temperature) might be required to completely vitrify the ware; the use of electrolytes and feldspar; shrinkage and density of cast pieces compared to those made in the manufacturer’s “normal” method (which is unspecified); optimal particle size; causes of pinholing; the use of revolving molds in casting;

applicable clay types; the effect of changes in slip viscosity; and techniques for measuring viscosity. As the article merely chronicled a discussion, no testing took place.

February 1926: Hewitt Wilson. “Monograph and Bibliography on Terra Cotta.” *Journal of the American Ceramic Society*.

This 43-page article described in detail the process of making terra cotta in 1926. Topics included the body (mixture, clay types, grog, salts & barium compounds, tempering, aging), plaster mold work, pressing and finishing, drying, slips and glazes (storage, preparation, spraying, temperatures, glaze fit, vitrification), and colors (common off-whites and polychrome). Although the author did not carry out any testing specifically for this article, solutions to many common problems in terra-cotta manufacturing were addressed. It was understood that these solutions were not revelatory, but rather spoke to common techniques employed in this era of the industry.

The author understood the effect weathering could have on a terra-cotta unit placed in a wall, but did not mention any predictive artificial weathering techniques.

This article also showed that the concept of “glaze fit” was well understood at the time. The author listed the most common defects and explained their causes:

- Thin glaze: This was a sprayed glaze problem, and was caused by too much water, which created a low viscosity of the glaze.
- Glaze cracking (crawling): This was the result of a glaze still in tension after fusion with the clay body. Glazes high in feldspar and clay content with low amounts of flux were prone to this defect. It could be prevented by additional flux and higher firing temperatures.
- Glaze peeling: The author noted that although peeling was often synonymous with shivering, there was in fact a difference. Peeling could be caused by a layer of soluble

salts, preventing proper glaze adhesion; this could be mitigated by the use of barium compounds. Tighter slips, created with increased clay or flux content, could be beneficial, but too tight of a slip might contribute to peeling. The author recommended minimally thin underslips. Glaze adhesion could be tested by tapping the surface of the ware with a chisel; if the glaze came off with clay body attached, then there was good adhesion.

- Crazing: Excessive tension in a glaze could cause cooling cracks, known as crazing. The cracks could be on the surface of the glaze, throughout the whole thickness of the glaze, or even extend into the clay body. The author noted that the lines usually intersect at 90 degree angles, but could also run parallel to one another.
- Shivering: Strong compressive strength in the glaze and weak tensile strength in the body could result in glaze separating from the body, sometimes with chips of the body still adhered to the glaze. The author noted that this frequently occurred at the edges or corners of pieces, where there was excess compressive force.

<u>July 1926</u>: D.F. Albery. “Grog for Terra Cotta.” *Journal of the American Ceramic Society*.

This article discussed the various issues around obtaining and using grog in terra-cotta manufacturing. Materials used in the industry for grog had been wide-spread, but were becoming more expensive and hard to find. The author advocated for the use of material specifically manufactured as grog, saying that benefits would include a steadier supply of material, uniform quality, and (eventually) a lower cost. He then went on to describe various possible methods of making grog.

July 1926: W.A. Hull. “An Attempt to Secure a Uniform Mixture of Fine and Course Particles in Grog from a Bin.” *Journal of the American Ceramic Society*.

This article noted that some methods of moving and storing grog inadvertently sorted it by size (examples of these methodologies involve using elevators or conveyors for movement). The author took it as a given fact that well-graded grog was beneficial in terra-cotta production, and thus designed a system for obtaining this easily. He experimented with chutes and troughs, and finally designed a method where the grog flowed, rather than dropped.

November 1926: W.J. Stephani. “Shivering.” *Journal of the American Ceramic Society*.

This article discussed attempts made to reduce shivering observed in both matte and glossy glazes on rounded, convex surfaces. The author experimented with reduced clay content in the glaze; a 5% reduction in the glossy glaze reduced shivering, but 10% was found to result in an unfused glaze. In the matte glaze, a 5% reduction was too much, resulting in an unhealed surface that did not fuse, but a 2.5% reduction produced satisfactory results. These methods were only effective with white glazes, as the changed clay content altered the colors of polychrome glazes.

The author also attempted to reduce shivering by altering the clay body content, which was ultimately determined to be more desirable. Test trials were conducted with various clay types, and one specific clay (unnamed in the study) was the most successful.

November 1926: Harry Spurrier. “Some Fundamentals of Terra Cotta.” *Journal of the American Ceramic Society*.

The author of this article noted that there was no consensus in the industry as to what made “the best terra cotta.” He then went on to analyze many previously fired bodies, and made various conclusions about the effects of certain factors. It was found that when coarse grog was used, voids would be left in the clay body after firing- microscopic analysis showed that these voids could reach 40%. The types of grog used varied widely, so it was difficult to draw conclusions as to what was actually beneficial. Because microscopic analysis was used, the author also described the process of cutting samples and mounting thin sections. It was also noted that internal strains in the terra-cotta body could be detected if the thin section curled.

The author also postulated that osmosis, or unequal pressures resulting in movement of water molecules, could be a cause of deterioration in terra cotta. However, he then went on to disprove this hypothesis.

The author also speculated that repeated freezing and thawing of units may result in weakening, even before the visual impact of deterioration (such as spalling) could be seen. To test this, the rate at which water flowed into terra-cotta pieces was measured. Pieces were then submerged in distilled water, frozen at -10°C, and then thawed. The rate of water flow was then measured again, and it was noted that this rate had increased since before the freezing procedure. Further repetitions yielded increasing rates. The author concluded from these experiments that freezing and thawing cycles were a cause of disintegration to terra cotta's internal structure.

**Note:** This article is similar, but less specific, to the procedures used for a contemporary freeze-thaw test.

<u>October 1927</u>: D.F. Albery. "Dry Grinding Clay to Eighty-Mesh." *Journal of the American Ceramic Society*.

In this article, techniques for grinding clay were given; first, however, the author explained the necessity for this process. Grinding clay eliminated the effect of copper pyrite in the clay, which could cause "specks, pop-outs, checking, etc." It was also stated that clay that had been ground yielded no difference in workability, drying, or warping. Various mills were used for the grinding, and the author described these processes.

February 1928: R.M. Murphy. “A Warpage Study of Terra Cotta Clays.” *Journal of the American Ceramic Society*.

The author’s terra-cotta production involved the use of many types of clay, which he cited as being common in the industry. However, it was noted that it was often difficult to determine the optimal proportions of these clays. In an effort to solve this problem, samples were made using various common clays and 33% grog (screen analysis was given). Nine by one by one inch bars were made and tested for warpage. Clays that were highly plastic yielded high amounts of dry warpage, but low warpage after firing; they also had the highest amounts of shrinkage during firing. The author did not ultimately conclude by suggesting proportions to be used, but mentioned the benefits of each type of clay: low-plasticity clays were useful for reducing shrinkage and warpage, sandy clays were important for reducing firing warpage and firing cracks, and high-plasticity clays created a tight firing quality and resistance to firing warpage.

May 1928: E.C. Hill. “Some Experiments on Terra Cotta Slips.” *Journal of the American Ceramic Society*.

This article examined the composition of various slips and compared this with their tendency to craze. The level of crazing and maturity of slips was determined using ink staining. It was found that high feldspar contents led to large cracks in the slip. When less than 50% clay was used, crazing usually occurred; this could be decreased by the addition of Cornwall stone. The author’s ultimate results were as follows: decreasing clay content effectively increased vitrification and decreased crazing, and increasing Cornwall stone content decreased cracking.

May 1928: H.G. Schurecht. “Methods for Testing Crazing of Glazes Caused by Increases in Size of Ceramic Bodies.” *Journal of the American Ceramic Society*.

In this article, the author showed that ceramicists of the 1920’s understood that crazing was caused by expanding terra cotta, and that the water content of the body versus glaze would be different. He said “It is a well-known fact that when a glaze contracts more than the body in cooling from the temperature at which it becomes rigid down to room temperature, it may craze.” The article was written to understand crazing that occurred after one or more years of natural weathering, not crazing that occurred in the kiln or during cooling.

A unit that was free from crazing after firing and then naturally weathered for twelve years was examined; after outdoor storage, it had become crazed. The expansion of this piece was measured while the piece was heated and cooled. Through a series of formulas, the expansion of the unit over the course of its lifetime so far was calculated. It was determined that expansion of the body under the glazed surface caused the cracks. Similar tests were done on an uncrazed piece that had also been weathered for twelve years, and it was found that the glaze and clay body expanded at similarly minimal rates.

These tests were furthered by creating and firing new samples and subjecting them to steam pressure (in an autoclave) of 150 psi for two hours, then drying them. It was found that the units which most resisted crazing had low porosity, low solubility in sulphuric acid, and “low ignition loss above 110°C.” The author suggested other methods of testing for crazing:

- “Absorption after two hours boiling and 24 hours soaking in water
- “Solubilities in sulphuric acid
- “Ignition losses above 110°C after outdoor storage for three years
- “Subjecting specimens to a steam pressure of 150 psi in an autoclave for 1 hour
- “Storing glazed specimens outdoors for three or more years”

The author stated that the results of three years of natural weathering and an autoclave test yielded similar results; he concluded that the autoclave was a good prediction of the tendency of a unit to craze.

**Note:** This article is later used as a guideline for the historic autoclave test.

July 1929: Raymond E. Birch. “The Relation of a Modulus of Grain Size to the Mechanical Strength of Sagger Mixtures.” *Journal of the American Ceramic Society*.

This article discussed the relationship of the strength of a terra-cotta unit and the “grain-size classification on modulus of grog.” After a series of tests, the author concluded that the best grog size was “classification modulus 1.5,” although it was difficult to translate what this number meant for the practical application of grog size and grading due to the complicated formulas.

April 1930: Gordon B. Richardson. “The Causes and Prevention of Bulging in Hand-Molded Refractory Shapes.” *Journal of the American Ceramic Society*.

This article discussed the tendency of the cut face of terra-cotta units to bulge. The cut face was defined as the side of the unit that was on the top, or open, side of a mold, which was placed down during drying. This defect could be caused by manufacturing methods, including uneven pressures, removal from the mold, or uneven drying. Clay type or mix could also be a factor. The author suggested carefully controlling these steps of the manufacturing to reduce bulging, although he claimed it may not be possible to completely eliminate this defect.

June 1930: H.G. Schurecht and G.R. Pole. “Method of Measuring Strains between Glazes and Ceramic Bodies.” *Journal of the American Ceramic Society*.

The author of this article understood that a glaze that was in tension would craze, but too much compression could lead to shivering. He stated that crazing required less tension than shivering required compression. In order to measure the amount of strain present in a clay body-glaze interface, a testing method was developed. A two-inch ring of terra cotta was fired and glazed on the exterior only. After firing, the piece was scored and cut along one side (creating a C-shaped piece). Reference marks would be placed on either side of this cut, and the distance measured. The author would then measure the change in this distance over time- if the glaze was in compression, the ring would curl inwards, and if it was under tension, the ring would unfurl and attempt to flatten itself. He noted that it was too complicated to measure the stresses within the glaze itself, however.

Matte glazes were often found to be in greater compression, as they showed less thermal expansion than other glazes that were tested. For this reason, they were less likely to craze than bright, polychrome (glossy) glazes.

The ring test was also repeated while soaking the piece in water for seven days, then drying. An autoclave steam pressure test was also conducted. These tests were conducted to measure expansion due to moisture.

**Note:** This article is later used as a guideline for the historic autoclave test.

<u>October 1930</u>: A.E.R. Westman and H.R. Hugill. “The Packing of Particles.” *Journal of the American Ceramic Society*.

This article was primarily about particle size and packing efficiency. It did, however, mention that it was a well-understood concept that how closely packed an aggregate (grog) was affected the physical properties of many materials, including terra cotta. The author gave many formulas and data points, including how to calculate “minimum percentage void” based on particle size distribution.

## 4. MATERIAL TESTING

In order to evaluate the effectiveness of historic weathering techniques and to compare them to contemporary methods, a set of tests was chosen to be completed. An emphasis was placed on selecting tests that differed from modern procedures in order to identify key differences in techniques and results. Historic tests considered were freeze-thaw cycles, a salt bath, use of an autoclave, and quenching. Due to the similarities to modern techniques (and lack of specific procedures given historically), the historic freeze-thaw test was not selected to be part of the thesis testing as an historic test. The historic salt bath contrasted from modern methods and was chosen to be carried out. Using an autoclave was a completely different approach from today's methods, and so it was also included. Quenching was not selected because it usually brought samples to complete failure within three cycles. The goal of this thesis was not to produce dramatic failure, but rather to weaken the pieces in an attempt to mimic natural weathering cycles and create measurable results.

The comparative contemporary tests chosen were the BRE crystallization test and the ASTM C67 section 9 freeze-thaw test. A group of samples was also chosen to remain as an unweathered control.

Although the BRE crystallization test is a British procedure, it was chosen due to the lack of a comparable American test. The closest procedure found was ASTM C67 section 11, titled "Efflorescence." However, this test was not selected for this thesis because it is not an artificial weathering test- rather it serves only to identify the presence of soluble salts that may exist within a unit by creating efflorescence on the unit's surfaces. It does not create cyclical stress upon the piece, as the BRE test does.

In order to evaluate the effectiveness of these tests and their propensity to encourage glaze failure, surface adhesion pull tests (ASTM D4541) were conducted on all samples (historic weathering, modern weathering, and control). Although the AIA File No. 9 Ceramic Veneer Specifications adhesion test was considered, its lack of quantifiable data made it unsuited for the needs of this thesis.[39] The AIA test consists of adhering a 1x1x4" bar to the glazed surface, then knocking or prying the bar off. A sample is considered to pass this test if the glaze does not pull off by itself, but rather takes clay body with it. Due to the pass/fail nature of this test, without measurable resistance, the ASTM procedure was chosen instead.

It should be noted that this thesis is not testing the resilience of each sample, but rather the effectiveness of the specific weathering techniques. To that end, it follows that samples from one building that were weathered in the autoclave, for example, were not compared to samples from a different building that were also subjected to the autoclave. Rather, autoclave samples were compared to controlled and weathered samples from the same building. It is the calculated effectiveness of these weathering techniques that was then compared building-to-building.

### 4a. The Samples

Samples were generously donated from Walter B. Melvin Architects and Essex Works Limited. All samples subjected to testing came from Manhattan buildings, except for one, located in Queens. All samples were likely hand-pressed or at least hand-finished, as indicated by finger markings on the back sides of the units and voids in the clay bodies. Information on the buildings from which the samples came, along with a visual analysis of the samples, follows. Colors of the glaze, clay body, and grog were matched using the Munsell Soil Color Book.

It must be acknowledged that all samples tested in this thesis were on buildings for many decades before their removal. Being located in New York City, they went through harsh cycles of winter weather. Traditionally, laboratory artificial weathering tests are performed on new

[39] Architectural Terra Cotta Institute. Public Works Specifications: Ceramic Veneer. A.I.A. File No. 9. October, 1961.

materials, as a means of predicting the durability of the piece. I originally obtained historic samples with the intent of tracing the evolution of clay body mixes; however, as my thesis topic evolved, my testing program changed with it. I decided to put the historic samples that had already been donated to me through the artificial weathering, taking care to put some of each sample through each type of test. This allowed me to effectively test the tests, not compare one building sample to another. I do not believe that the previously weathered state of my samples was a hindrance to my testing program- it merely weakened the pieces, requiring that less work be done by the artificial weathering I carried out. It is understood that samples made by the same manufacturer on the same building may still weather differently- terra cotta is a material that is difficult to fully understand and predict; despite the fact that it is artificially made, it can still vary widely in its physical properties. It is for this reason that as many samples as possible were obtained, in order to average results within sample groups.

Sample A*: 135 Central Park West: The Langham*

- Architect: Clinton & Russell
- Date of construction: 1904
- Landmark: Yes
- Sample location: Dormer soffit


Figure 13 (left): The Langham, 135 Central Park West. Source: www.nyc-architecture.com
Figure 14 (right): Sample A location on building, soffit. Source: Doug Schickler

Sample A specimens have a combed surface, glazed with a matte glaze of 10YR 7/2 “light gray” with 10YR 3/3 “dark brown” specks. Crazing is difficult to see, but was identified under a microscope after artificial weathering.[40] The clay body is 2.5Y 8/2 “white” with grog of 10YR 8/6 “yellow,” bright white (not in soil chart), and 2.5Y N4/0 “dark gray” colors. Grog pieces ranged in

[40] See note 48 on page 88.

size from 0.11mm to 3.28mm across, and voids ranged from 0.09mm to 1.05mm wide and 0.18mm to 4.52mm long.

Figure 15: Samples from group A before testing

Figure 16 (left): Example of sample A glazed surface
Figure 17 (right): Example of sample A clay body and combed surface

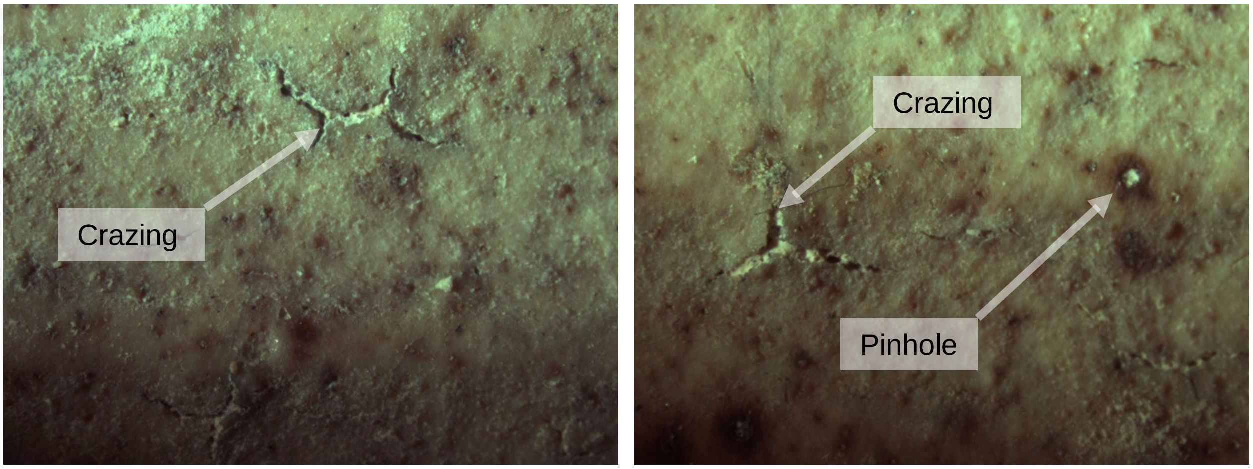


Figure 18: Sample A glaze

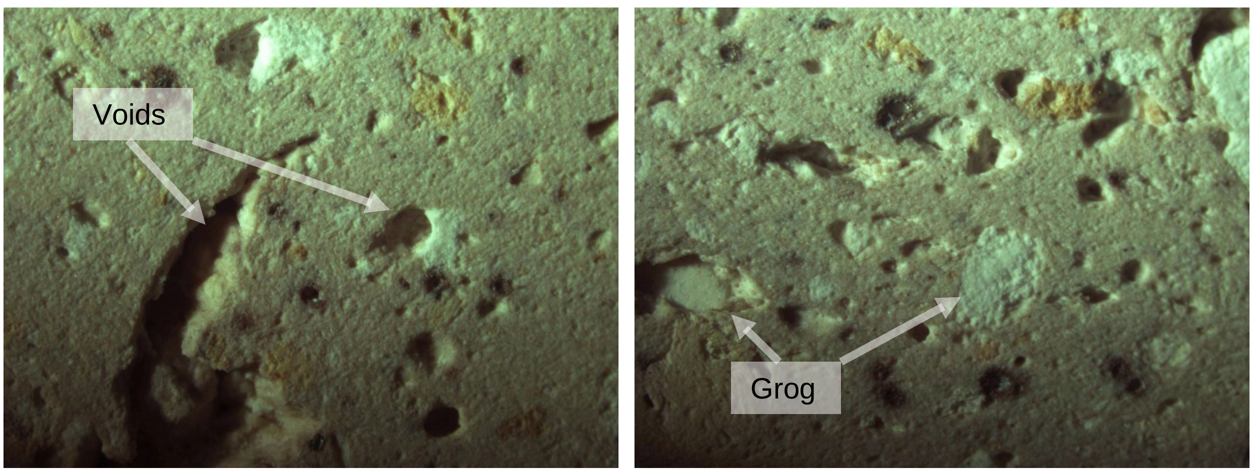


Figure 19: Sample A clay body

*Sample B: 998 Fifth Avenue*

- Architect: McKim, Mead, & White
- Date of construction: 1911
- Landmark: Yes
- Sample location: Cornice


Figure 20 (left): 998 Fifth Avenue. Source: *Real Estate Record and Builder's Guide* 96.2470 (1915): 117.
Figure 21 (right): Sample B location on building. Source: Walter B. Melvin Architects, LLC


Figure 22: 998 Fifth Avenue cornice. Source: Walter B. Melvin Architects, LLC

Sample B specimens are glazed with a matte, 2.5Y N7/0 “light gray” glaze, with no specks or mottling. Most areas show signs of crazing. The clay body is 5Y 8/3 “pale yellow,” with grog of 2.5Y 8/6 “yellow,” 2.5Y N3/0 “very dark gray,” and 2.5Y 8/4 “pale yellow” colors. Grog pieces ranged in size from 0.29mm to 2.21mm across, and voids ranged from 0.22mm to 0.62mm wide and 0.70mm to 2.07mm long. Samples which were subjected to testing were broken on-site by construction workers- only a single piece was cut for cross-sectional examination.


Figure 23: Samples from group B before testing

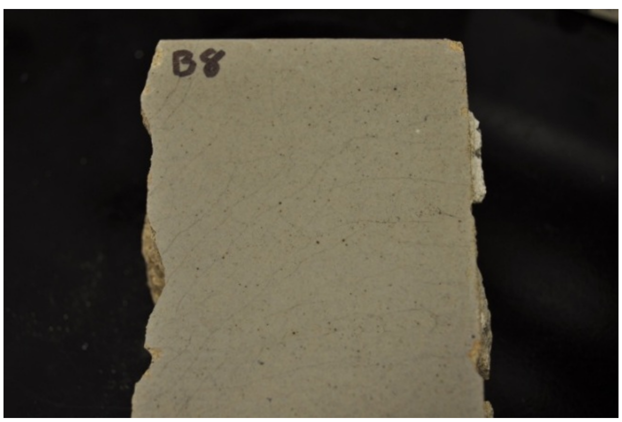



Figure 24 (left): Example of sample B glazed surface
Figure 25 (right): Example of cut sample B clay body

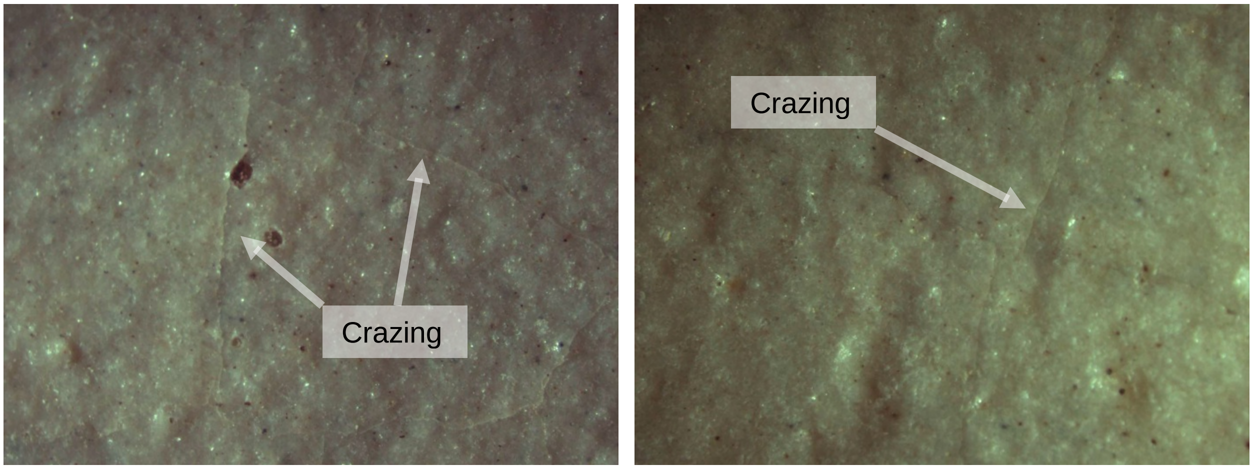


Figure 26: Sample B glaze

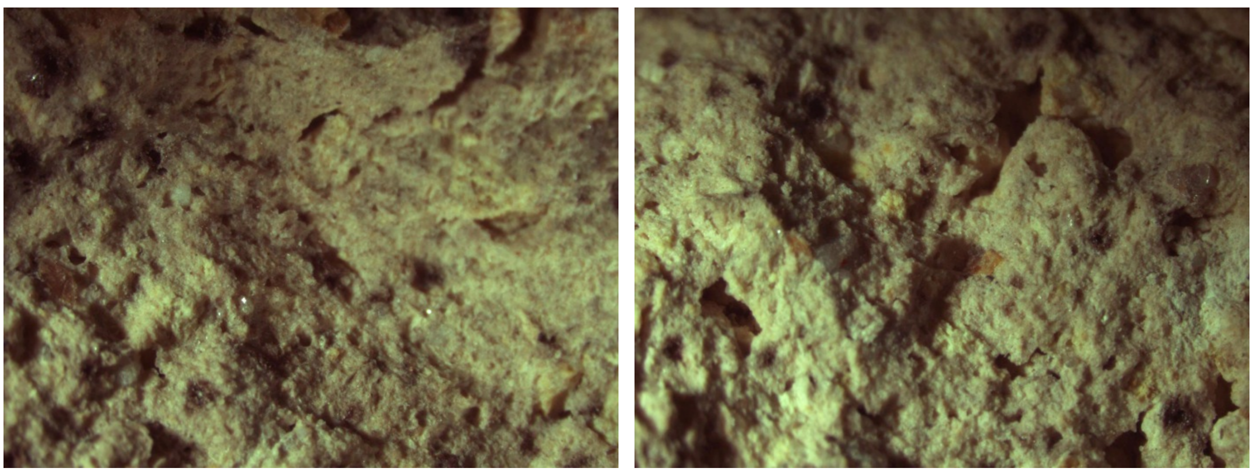


Figure 27: Sample B clay body- broken, uncut surfaces (microscope with camera attachment was not available after a sample was cut for examination)

*Sample C: 57 West $57^{th}$ Street (1400-1410 Sixth Avenue)*

- Architect: Warren & Wetmore
- Date of construction: 1927
- Landmark: No
- Sample location: Spandrel

Figure 28: 57 West 57th Street

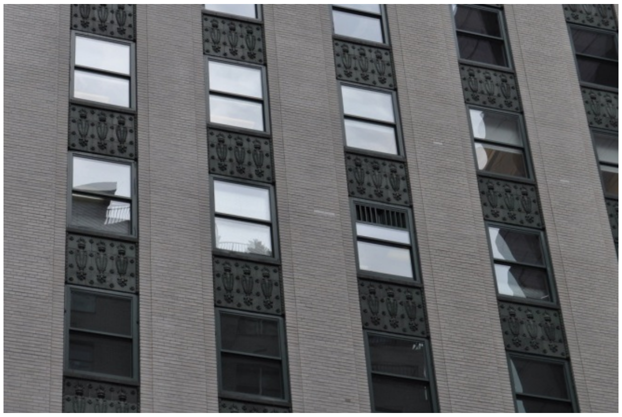



Figure 29 (left): Sample C location on building
Figure 30 (right): Sample C location on building, detail. Source: Doug Schickler

Sample C specimens have a glossy 5G 4/8 “bright green” glaze.[41] The glaze is speckled with a 5Y 2.5/1 “black” that mottles the surface- this may have been applied by spraying action over the green layer of glaze. On unexposed sides of sample C specimens, the green is brighter. Faint crazing can be seen, and the cracks are spread wide apart. The clay body is 10YR 8/4 “very pale brown,” with grog of 7.5YR 5/6 “strong brown,” 2.5Y 8/4 “pale yellow,” and bright white (not in soil chart) colors. Grog pieces ranged in size from 0.11mm to 2.53mm across, and voids ranged from 0.06mm to 1.97mm wide and 0.29mm to 12.48mm long.

[41] This designation came from the Munsell Book of Color, not the Soil Chart.

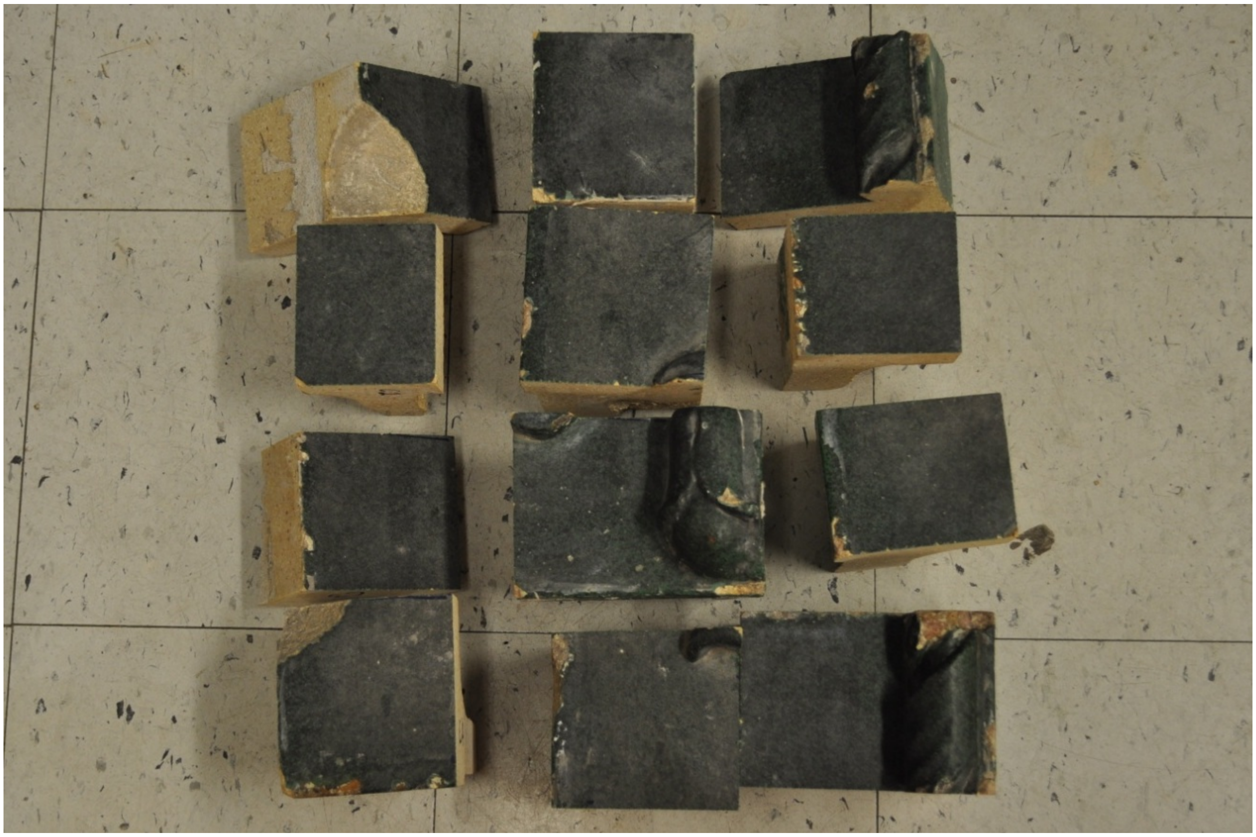
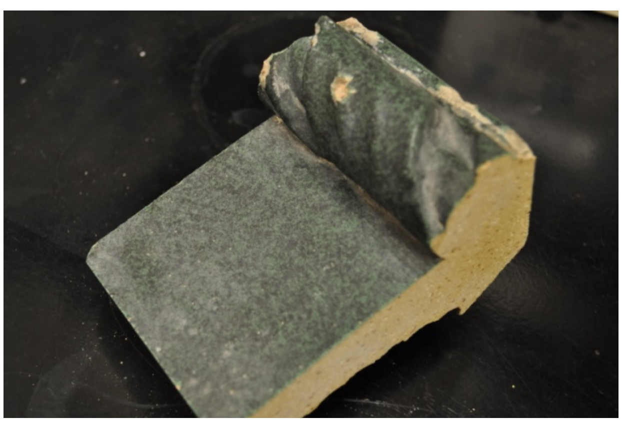


Figure 31: Samples from group C before testing



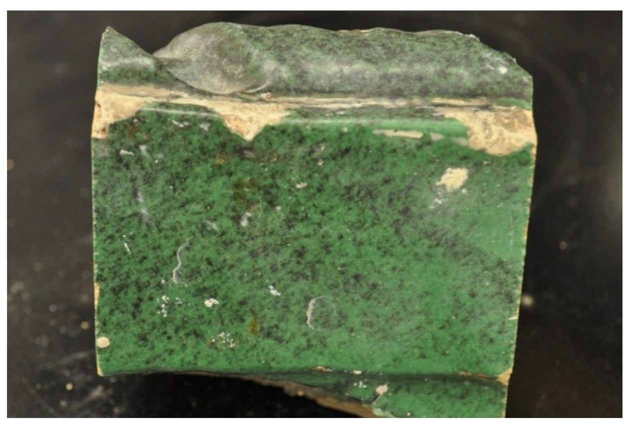


Figure 32 (left): Example of sample C glazed surface: top
Figure 33 (right): Example of sample C glazed surface: side

Figure 34: Example of sample C clay body

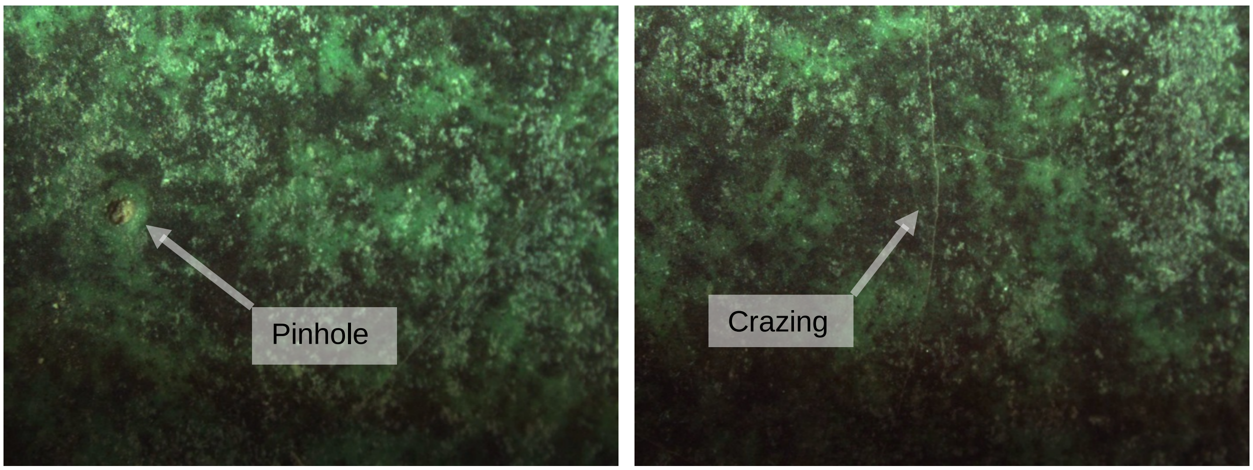


Figure 35: Sample C glaze

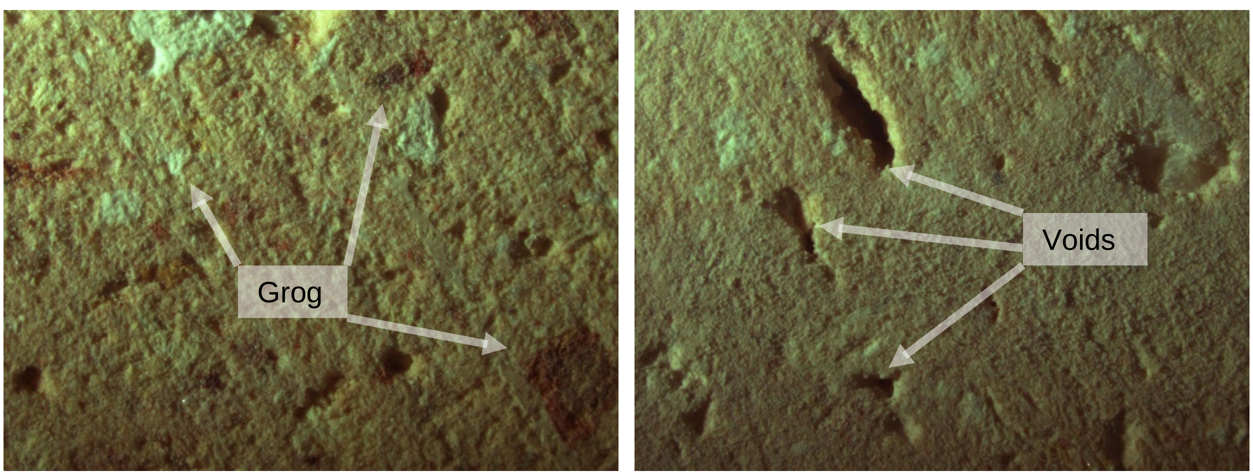


Figure 36: Sample C clay body

*Sample D: 35-55 76$^{th}$ Street (Hawthorne Court), Jackson Heights, Queens*

- Architect: George H. Wells
- Date of construction: 1921-22
- Landmark: contributing building in Jackson Heights Historic District
- Sample location: Cornice

Figure 37 (left): 35-55 76th Street, Jackson Heights, Queens
Figure 38 (right): Sample D location on building. Source: Doug Schickler

Sample D specimens have a matte 2.5Y 7/2 “light grey” glaze with 10YR 2.5/1 “reddish black” specks. Due to soiling in roughly half of the samples, the glaze appeared anywhere from a 5Y 6/2 “light olive gray” color to 2.5Y N3/0 “very dark gray.” The cause of difference in this layer of soiling is unknown- it could have been created by a previous application of an acid wash, for example; the soiling could not be removed by light scrubbing. Samples were divided such that each weathering test was performed on a dirty and clean sample. Crazing was present. The sample D clay body is 10YR 8/3 “very pale brown” with grog of 2.5Y 8/4 “pale yellow,” 2.5Y 7/8 “yellow,” and 10YR 8/2 “white” colors. Grog pieces ranged in size from 0.17mm to 2.47mm across, and voids ranged from 0.07mm to 1.61mm wide and 0.31mm to 22.32mm long.


Figure 39: Samples from group D before testing

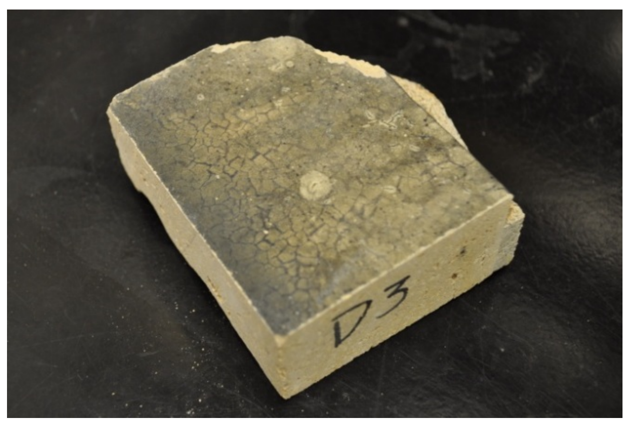
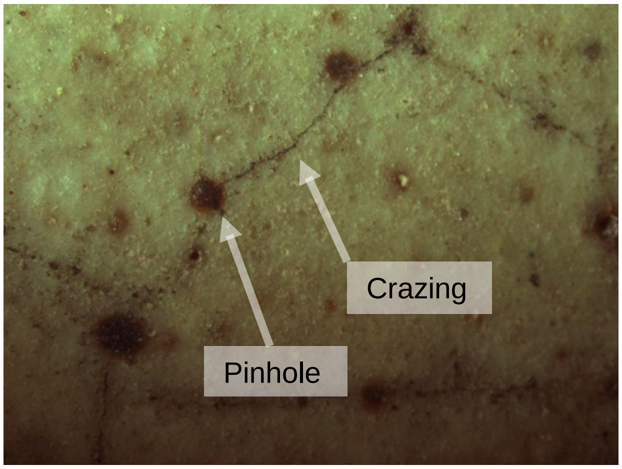




Figure 40 (left): Example of sample D clean glazed surface
Figure 41 (right): Example of sample D dirty glazed surface

Figure 42: Example of sample D clay body



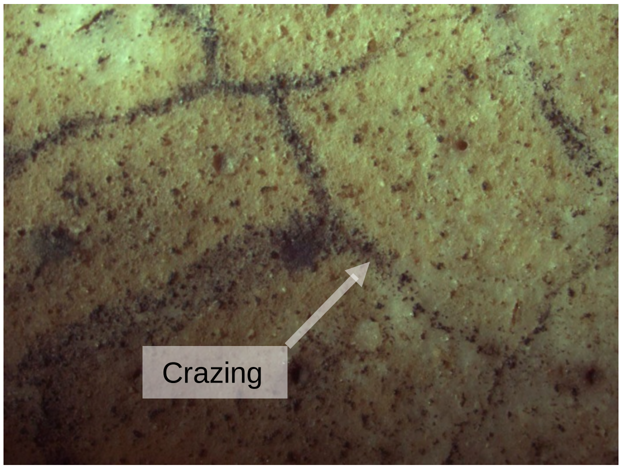
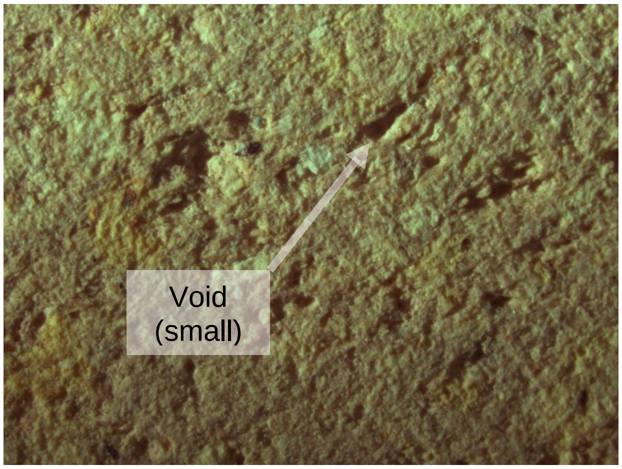




Figure 43: Sample D glaze



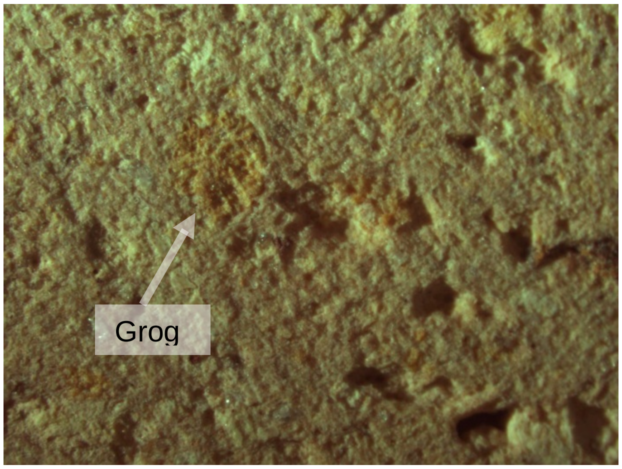




Figure 44: Sample D clay body

*Sample E: 245 West 107th Street*

- Architect: Sugarman & Berger
- Date of construction: 1928
- Landmark: No
- Sample location: Balconette

Figure 45 (left): 245 West 107th Street
Figure 46 (right): Sample E location on building. Source: Doug Schickler

Sample E specimens have a matte 2.5YR 6/6 “light red” glaze with 2.5YR N2.5/0 “black” specks. Crazing lines are present. The clay body is 10YR 8/3 “very pale brown,” with grog of 2.5Y 8/4 “pale yellow,” bright white (not in soil chart), and 2.5Y 7/8 “yellow” colors. Grog pieces ranged in size from 0.13mm to 2.11mm across, and voids ranged from 0.13mm to 1.00mm wide and 0.33mm to 18.54mm long.

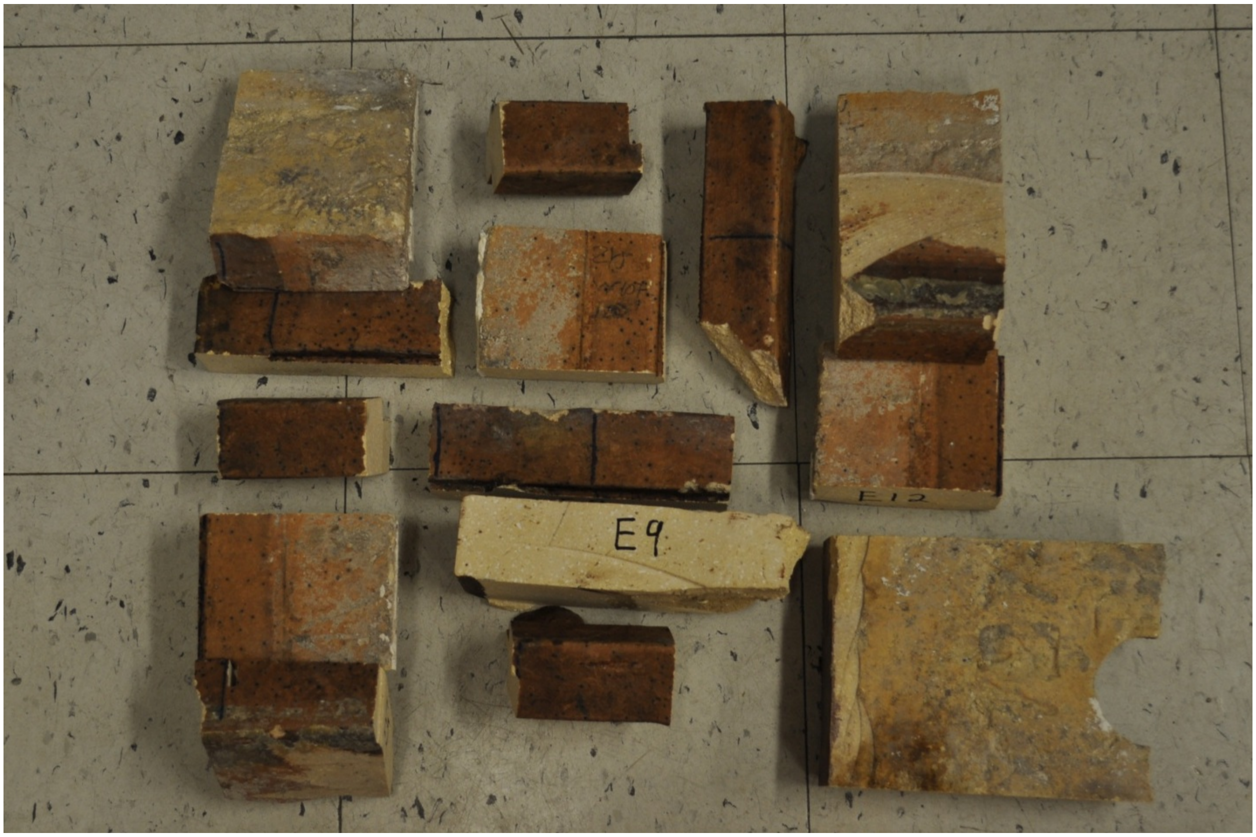



Figure 47: Samples from group E before testing

Figure 48 (left): Example of sample E glazed surface
Figure 49 (right): Example of sample E clay body

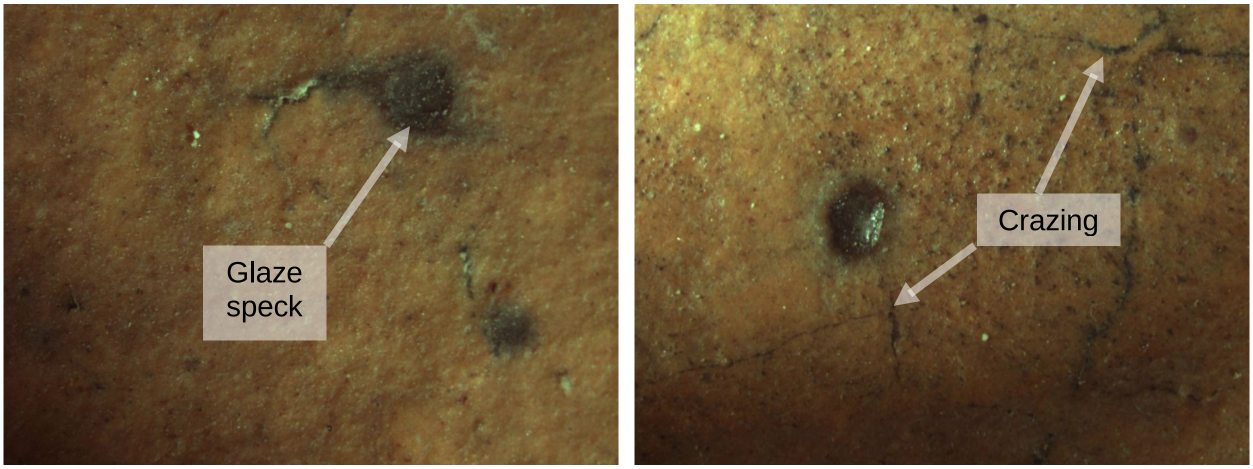


Figure 50: Sample E glaze

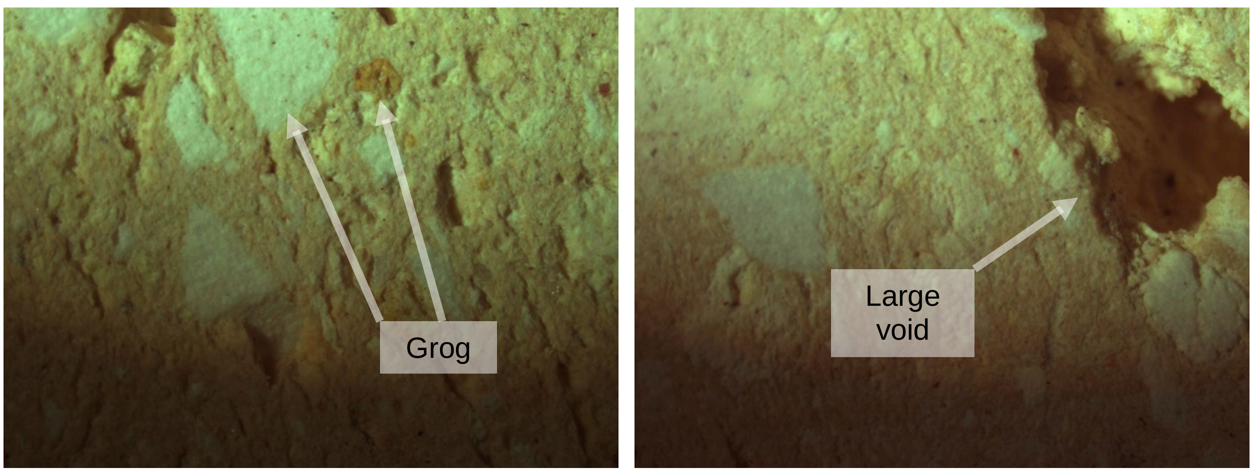


Figure 51: Sample E clay body

### 4b. Sample preparation

Before testing could begin, samples had to be cut into testable-sized pieces. Due to the nature of the tests to be performed, only flat surfaces of at least 3" in length could be used. All pieces of terra cotta (except group B) were brought to the Carleton Laboratory on the Columbia University campus. Having been marked with a permanent marker, the samples were cut by a machinist in the lab who used a wet saw. Samples from 998 Fifth Avenue were not cut with a wet saw, as the pieces were broken on location at the building by construction workers (except for one piece, which was cut for cross-sectional examination). It is possible that the action of breaking the pieces could have caused microcracks in their surface, creating a situation with a stronger possibility of failure. If this is the case, however, all samples were affected relatively equally.


Figure 52: A sample being cut on the wet saw

After the samples were cut, they were scrubbed in lukewarm water with a soft synthetic bristle scrub brush to remove dirt and loose debris. They were then briefly dried in an oven at 217°F for about an hour. Thorough drying was not deemed necessary because all samples would be submerged in water upon testing, and weight was not being used as an evaluation of degradation.

Samples were then labeled with permanent markers. Labels were assigned based on sample letter and the piece's number (example A1, A2, A3, B1, B2, B3, etc.). Because the donated pieces of terra cotta were of varying sizes, some sample groups encompassed more sample pieces than others. Samples from each building were evenly distributed between all tests to be carried out. For this reason, sample numbers do not correspond to specific tests.

| **Boiling Salt Bath** | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| A1 | A2 | A3 | A4 | A5 | A6 | A7 | A8 | A9 |
| B1 | B2 | B3 | B4 | B5 | B6 | B7 | B8 | B9 |
| C1 | C2 | C3 | | | | | | |
| D1 | D2 | | | | | | | |
| E1 | E2 | E3 | | | | | | |
| **Autoclave** | | | | | | | | |
| A10 | A11 | A12 | A13 | A14 | A15 | A16 | A17 | A18 |
| B10 | B11 | B12 | B13 | B14 | B15 | B16 | B17 | B18 |
| C4 | C5 | C6 | | | | | | |
| D3 | D4 | | | | | | | |
| E4 | E5 | E6 | | | | | | |
| **Freeze-Thaw** | | | | | | | | |
| A19 | A20 | A21 | A22 | A23 | A24 | A25 | A26 | A27 |
| B19 | B20 | B21 | B22 | B23 | B24 | B25 | B26 | B27 |
| C7 | C8 | C9 | | | | | | |
| D5 | D6 | | | | | | | |
| E7 | E8 | E9 | | | | | | |
| **Crystallization** | | | | | | | | |
| A28 | A29 | A30 | A31 | A32 | A33 | A34 | A35 | A36 |
| B28 | B29 | B30 | B31 | B32 | B33 | B34 | B35 | B36 |
| C10 | C11 | C12 | | | | | | |
| D7 | D8 | | | | | | | |
| E10 | E11 | E12 | | | | | | |
| **Control** | | | | | | | | |
| A37 | A38 | A39 | A40 | A41 | A42 | A43 | A44 | A45 |
| B37 | B38 | B39 | B40 | B41 | B42 | B43 | B44 | B45 |
| C13 | C14 | C15 | | | | | | |
| D9 | D10 | | | | | | | |
| E13 | E14 | | | | | | | |

## 4c. Testing procedures- weathering

*4ci. Historic boiling salt bath*

This testing procedure was based on the following article: Minton, R. H. "The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies." *Journal of the American Ceramic Society* 1.3 (1918).

1. Prepare a solution of 14% sodium sulfate, using sodium sulfate anhydrous and distilled water.
    - Since the historic test did not indicate the amount to be added, it was decided to follow the same amount as given in the modern crystallization test.
2. Heat solution on a hot plate in a large enamel pot until boiling. Use a lid to prevent evaporation and to reduce the amount of time required to reach a boil.
3. Add all samples to water and return to a boil. Once a boil is regained, leave samples in solution for 3 hours. Add solution as needed to keep samples submerged.
4. After 3 hours of boiling, remove samples and let cool at air temperature (69°F, 20-42% relative humidity) for 21 hours. Stand samples such that glazed surface to be later tested for adhesion is not on bottom of sample.
    - Although the historic test dried the samples for 48 hours, 21 hours were used for this test for the following reasons:
        - The historic literature stated that “no apparent effect of this treatment was observed,” and yet the samples tested for this thesis displayed dramatic visual differences very quickly- both on the clay body and glazed surfaces- as will be discussed in observations Chapter *4ei*. Therefore, the worry that a shorter cycle would not allow for sufficient salt crystallization was determined to be accounted for, since abundant crystallization

occurred and visual effects were seen through crazing lines on the glazed surfaces.

- Additionally, a 24-hour cycle (rather than the prescribed 51) allowed for the test to go through the same number of cycles as other tests that were being performed, allowing for all tests to stay on the same schedule and have easily comparable results.

5. Repeat process of boiling and air drying for 15 days. When a day of testing is skipped, hold samples at room temperature.
   - Historic tests did not dictate the number of cycles to be completed; previous ceramicists ran these tests until visual signs of deterioration were present (15-34 days, depending on the sample). 15 days were decided for this test, in order to be comparable with the prescribed length of the BRE crystallization test.

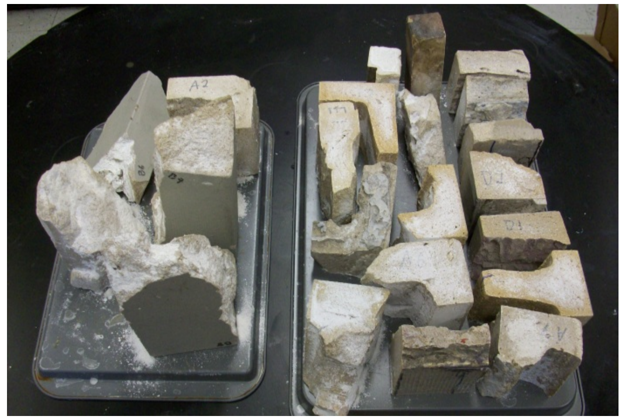


Figure 53 (left): Boiling test in progress
Figure 54 (top right): Samples drying at room temperature
Figure 55 (bottom right): Samples boiling in salt bath

*4cii. Historic autoclave*

The autoclave in the Biomedical Engineering department at Columbia University was utilized for these tests. An autoclave is an oven-like chamber that is capable of producing high pressure and/or temperature environments. Testing procedures used were based on test in the following articles: Schurecht, H.G. “Methods for Testing Crazing of Glazes Caused by Increases in Size of Ceramic Bodies.” *Journal of the American Ceramic Society* 11.5 (1928) and Schurecht, H.G and G.R. Pole. “Method of Measuring Strains between Glazes and Ceramic Bodies.” *Journal of the American Ceramic Society* 13.6 (1930).

1. Insert samples into chamber of autoclave. Use plastic containers as necessary to prevent abrasive damage to interior metal surfaces.
2. Firmly lock door and set test to run at 250°F and 20 pounds per square inch for 60 minutes.
    - Although historic tests prescribed using 150 psi, 20 psi was the highest pressure available due to the physical limitations of the building’s steam supply. The implications of this difference will be discussed in the results.
3. Because the steam application leaves the samples wet, dry them in an oven at 217°F for 4 hours.
4. Test is only performed once, and does not need to be repeated cyclically.
    - A single autoclave test was said to closely correlate with the behavior of a terra cotta unit that was weathered naturally for one to three years.


Figure 56 (left): Samples in the autoclave, before procedure
Figure 57 (right): Autoclave door shut during procedure

Figure 58 (left): Autoclave control panel during procedure

*4ciii. Freeze-thaw*

ASTM C67 section 9 was followed for this test.[42] The following historic articles also followed similar procedures, but gave less specific directions: Minton, R. H. "The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies." Journal of the American Ceramic Society 1.3 (1918) and Spurrier, Harry. “Some Fundamentals of Terra Cotta.” *Journal of the American Ceramic Society* 9.11 (1926). Due to the unspecific nature of these articles’ procedures, the freeze-thaw test was chosen to be considered as a contemporary test.

1. Submerge samples in a tank of distilled water, referred to as the thawing tank, for 4 hours.
2. Move samples to freezing trays with ½ inch of distilled water. Space samples approximately ½ inch apart, and freeze for 20 hours at 9°F.
    - Disposable aluminum baking pans were used as freezing trays.
    - Testing procedures state that the “head face” should be facing down in the tray; this was interpreted to mean that an unglazed side should be defined as the head face, leaving the outward-facing primary glazed surface only partially submerged in the water.
3. After the freezing period, remove samples from trays, using a hammer as necessary to break the ice. Place samples back in thawing tank.
4. Repeat this cycle of freezing and thawing for 25 days. If a day of testing is skipped, keep samples in freezer.
    - The testing procedure prescribed by ASTM specifies repeating these cycles until failure, or for 50 days- whichever occurs first. 25 cycles were completed for this thesis due to scheduling constraints. Additionally, the goal of this testing was not

[42] ASTM Standard C67-11, 2011, “Standard Test Methods for Sampling and Testing Brick and Structural Clay Tile,” ASTM International, West Conshohocken, PA, 2011, DOI: 10.1520/C0067-11, www.astm.org.

to push a piece to complete failure- but rather to merely weaken it, with the hopes of measuring that weakening power with the adhesion test.

Figure 59 (left): Samples in freezer in standing trays
Figure 60 (right): Detail of samples in freezer in standing trays

Figure 61: Samples submerged in thawing tank

*4civ. Crystallization*

The BRE Crystallisation Test procedures were followed for this test.[43]

1. Submerge samples in 14% solution of sodium sulfate anhydrous made with distilled water at room temperature.
    - After 4 days of testing, deionized water was used to prepare the solution instead, due to its ready availability in Columbia University's conservation science lab.
2. After samples have soaked for 2 hours, move them to an oven at 217°F, arranged such that surfaces to be tested for adhesion are on the top or side of the sample, but never the bottom.
3. After 16 hours, remove samples from oven to cool at room temperature, or 20-42% relative humidity and an average of 69°F.
4. Repeat this process daily with freshly made solution for 15 days. If a day of testing is skipped, hold samples in the oven.

[43] Ross, K. D. Durability Tests for Building Stone. Rep. no. 141. Garston, Watford: Building Research Establishment, 1989.


Figure 62 (left): Samples in oven
Figure 63 (top right): Samples arranged in oven
Figure 64 (bottom right): Samples in salt bath

*4cv. Control*

One group of samples was left unweathered. These pieces were left at room temperature during the duration of the artificial weathering tests, in a room of 20-42% relative humidity and an average of 69°F.

**4d. Testing procedures- surface adhesion**

All samples put through historic and modern weathering tests, and the samples left as control, were subjected to a surface adhesion test using an Elcometer 106 Pull Off Adhesion Tester, which can apply a range of 0-1,000 pounds per square inch. ASTM D4541 procedures and the Elcometer 106 instruction manual were followed for this test.[44]

1. Thoroughly dry samples until constant weights are reached, ensuring a loss of all moisture.
2. Clean surfaces to be tested with acetone to remove dirt and/or salts. Also clean bottom surface of dollies.
3. Araldite 2011 A/B epoxy adhesive is recommended for contact between metal and glazed ceramic.[45] Mix two-part epoxy according to directions, and apply thinly to dolly.
    - Samples from group A, which had combed surfaces, required extra epoxy to ensure full contact between glaze and dolly.
4. Apply dolly to terra cotta surface, pressing firmly and taking care not to slide dolly. Ensure that terra cotta surface is level to prevent slippage, and allow epoxy to cure for at least 15 hours.
5. To begin surface adhesion test, place bearing ring, which is included with Elcometer 106, concentrically around the dolly.
6. Reset the Elcometer, if necessary, to lower the claw mechanism and calibrate the force readout to 0 psi.
7. Slide the Elcometer onto the sample, such that the three feet of the unit rest on the bearing ring, and the claw engages the dolly. (See photos below.)

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[44] ASTM Standard D4541-09, 2009, "Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers," ASTM International, West Conshohocken, PA, 2010, DOI: 10.1520/D4541-09E01, www.astm.org.
[45] Epoxy advice was obtained from a Technical Adhesive Representative at Industrial Sales Associates, Inc. (www.adhesivehelp.com)

8. Turn the wheel at the top of Elcometer, tightening the mechanism, which in turn raises the claw. Turn the wheel as smoothly and continuously as possible, because sustained pressure at a lower level may have a similar effect as one of a slightly higher level. A continuous motion ensures an accurate reading.
9. When the glaze fails, the dolly will pull away from the surface with a loud pop. Record the psi on the gauge readout, and set the piece aside.
    - The force applied is indicated in 100-psi increments as a pin is dragged along the force readout scale while the device is employed. Readouts were estimated in 25-psi increments.
10. Repeat these steps as necessary on all samples, noting observations and data in a table.[46]
11. In order to reuse a dolly, heat it at ±350°F for 4-5 hours. At this point, a sharp object such as a scalpel or blade can be used to pry the clay body and epoxy off the dolly's surface. Use 100 grit, then 150 grit sandpaper to smooth the surface of the dolly before reuse.



Figure 65 (left): The Elcometer 106, bearing ring, and loading dolly
Figure 66 (right): Model information at the top of the Elcometer

[46] On some samples, two to three surfaces were tested to maximize the number of data points attained. These surfaces were never on the same plane, to prevent stress from the adhesion test on one surface affecting the results on another.

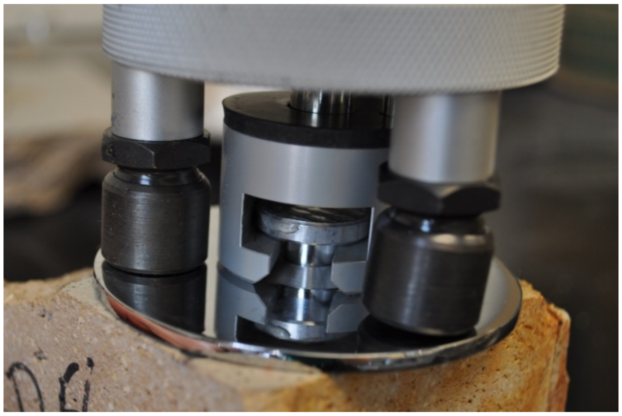




Figure 67 (left): The claw and feet of the Elcometer, as loaded onto the dolly and bearing ring
Figure 68 (right): The force readout scale, in lb/in$^2$ x 100

At the beginning of testing, a number of dollies were accidentally epoxied onto surfaces upside-down, preventing the Elcometer claw from properly fitting.[47] In order to remove these dollies from the samples, they were heated at apprximately 350°F for 1 hour, which slightly softened the epoxy. The dollies could then be pried off of the terra cotta surfaces with pliers. When the dollies were re-adhered, a new location was selected, in order to prevent overlap with any epoxy remnants on the surface. In the process of prying off the dollies, however, three samples reached failure: B10, E4, and E5.

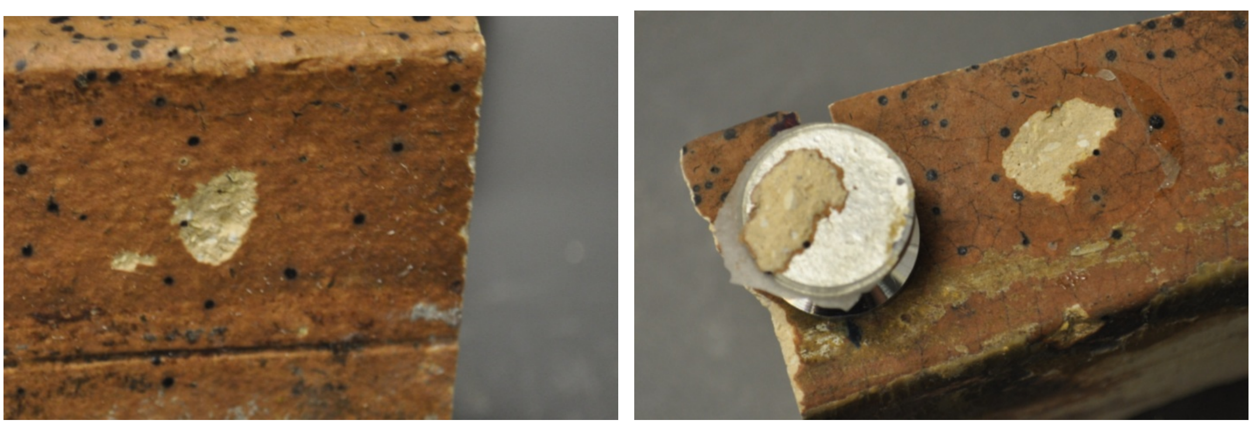


Figure 69: Failure by manual prying on samples E4 and E5

[47] The heated and re-epoxied samples were: A10, A11, A12, A13, A14, A15, A16, A17, A18, B10, B12, B13, B15, C4, C5, C6, D3, D4, E4, E5, and E6.

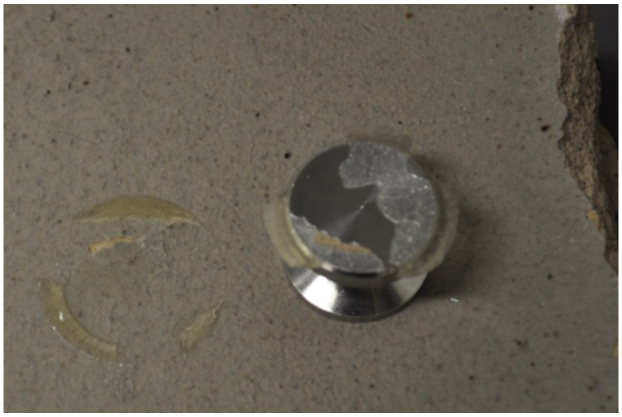


Figure 70: Failure by manual prying on sample B10

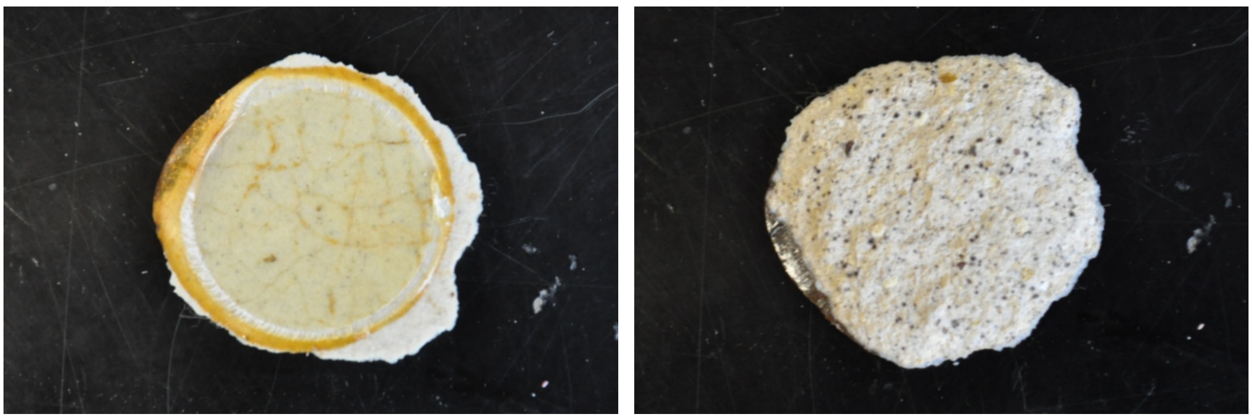


Figure 71: Material removed from a dolly, including intact epoxy, glaze, and clay body, after a successful adhesion pull test

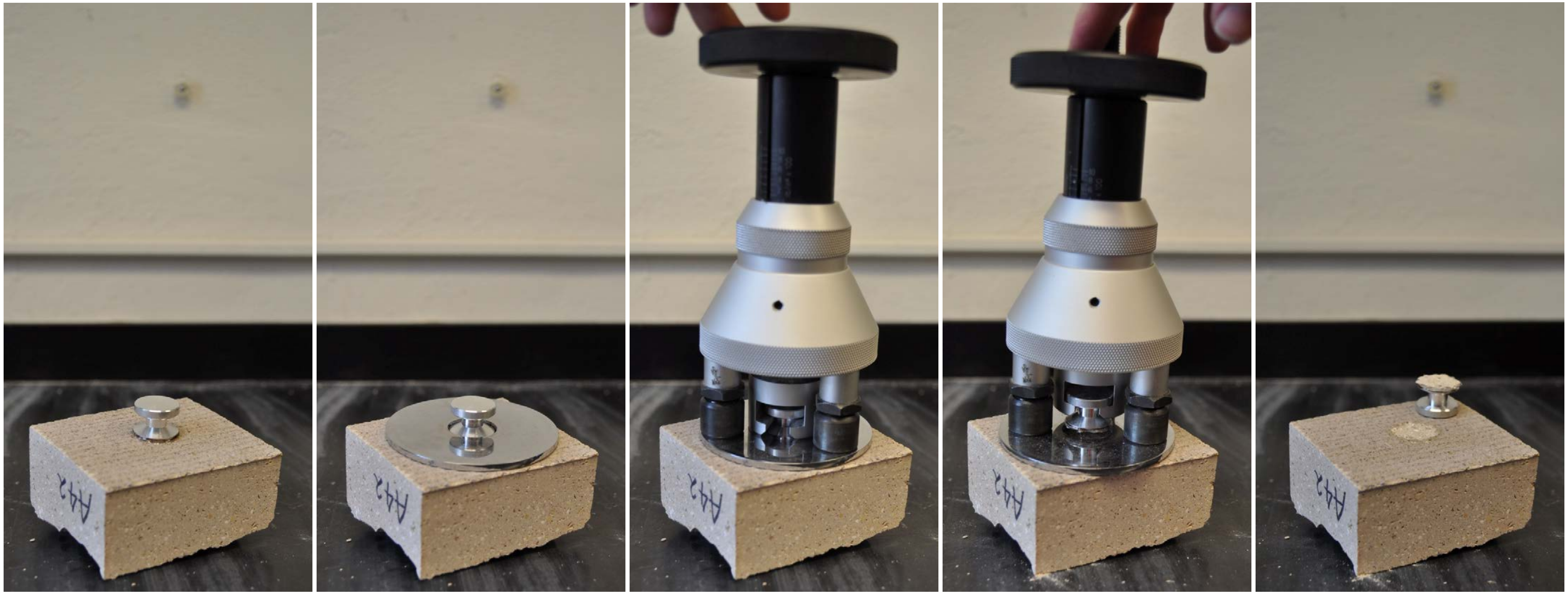


Figure 72: Sequential images of the adhesion test being performed on a sample. (L-r): Dolly adhered to surface, bearing ring added, Elcometer mounted, Elcometer deployed, dolly separated from sample

## 4e. Testing observations

*4ei. Historic boiling salt bath*

Upon air drying, all samples subjected to the boiling salt bath test had sodium sulfate deposits on their unglazed surfaces, in the form of a light powder. This was far more evident on sample group B; it is unknown if the abundant salts were caused by the material make-up of this sample, or simply because the surfaces were roughly broken, not cut like other sample groups. Historic tests never mentioned these deposits in descriptions of the experiments carried out, but the reason for their presence is easily understood.

Figure 73: Typical sodium sulfate deposits

During testing, the visual appearance of crazing patterns was heightened on most samples. After approximately 3 days of testing, some surfaces on sample B pieces had traces of salt deposits that outlined previously existing crazing lines. These were not limited to flat surfaces, but also sometimes appeared on curved surfaces. Rarely was an entire surface covered in this distinctive pattern; the occurrences were usually patchy in their placement and density, in according with the crazing. These patterns became increasingly pronounced as testing continued. Salt deposits were also concentrated around areas where the glaze had been previously chipped, revealing bare clay body.

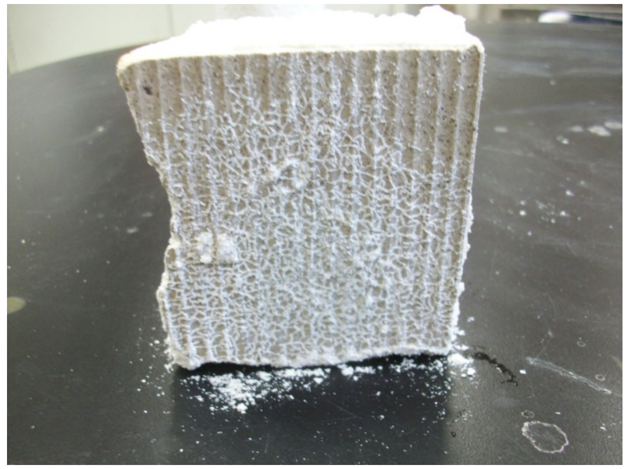



Figure 74: Salt patterns over crazing lines on sample B3
Figure 75: Salt patterns over crazing lines on sample A4


Figure 76 (left): Sample B8 after 15 testing cycles
Figure 77 (right): Salt deposits concentrated around clay body exposures

Similar patterns were also noticed on a sample from group A after 9 days. The pattern was very densely spaced. This was notable due to the combed surface of this piece, and the delayed time required to see this evidence compared to other samples.[48] After 10 days, these patterns also developed on samples from groups D and E.

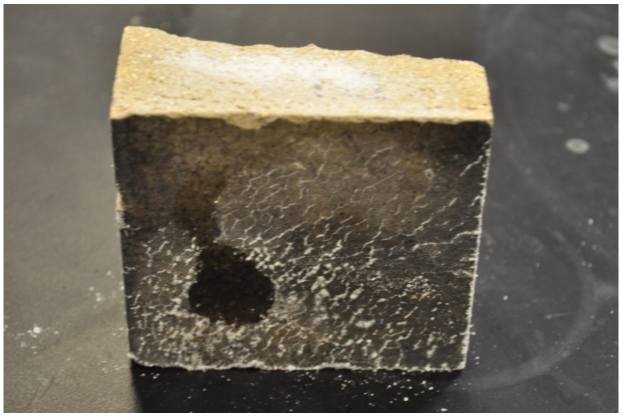



Figure 78 (left): Salt-traced crazing patterns on sample from group D
Figure 79 (right): Salt-traced crazing patterns on sample from group E

On pieces from sample group C, upon removal from the boiling water, the crazing pattern was shown in steam on the surface. There was a difference in the way that the vapor evaporated off the surface between the solid, glossy glaze and areas that had crazed. The specific nature of this behavior is not understood, but is useful in determining the exact locations of crazing lines, which were difficult to identify before testing. This layer of steam would disappear as the sample cooled. After 10 days, this pattern could still be seen in salt deposits on the surface; the pattern was reversed from those previously discussed with other sample groups.

[48] It is worth noting that I did not originally know with normal visual inspection that pieces from the sample A group exhibited crazing. Upon the appearance of this salt pattern, however, I examined a control sample from the same group under a microscope, revealing crazing cracks. The sample description sections were thus updated accordingly with photo micrographs. Without this test, the crazing may have gone unnoticed.


Figure 80: Sample from group C being removed from the boiling salt bath

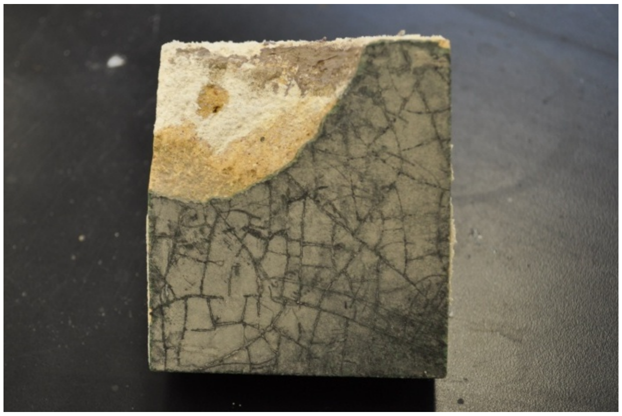
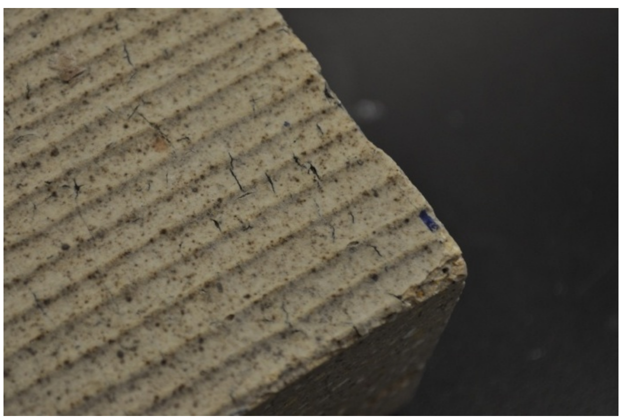


Figure 81: Sample from group C after drying

The crazing lines on sample group A were easier to identify after 10 days of testing; the cracks were darker and more visible against the light glaze. It was unclear if the cracks were widened by testing, or simply rendered darker after the procedure. Similarly, crazing lines on samples from group B were also more evident after roughly 10 days of testing.



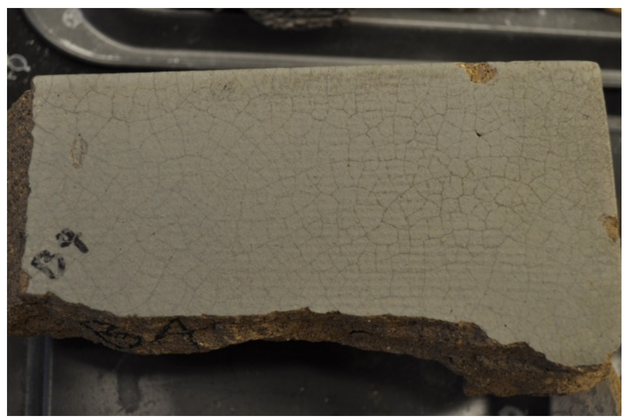


Figure 82 (right): Evident crazing lines on sample from group A after being removed from water
Figure 83 (right): Evident crazing lines on sample B7 after drying

Upon testing completion, a small amount of sediment remained in the bottom of the pot used for boiling. It is unknown if this was due to sample disintegration, or simply bits of clay body that were already loose before testing, which were knocked off of the samples during the procedure.

Figure 84 (left): Sediment in the bottom of the pot after testing
Figure 85 (right): Sediment seen on a sample surface

*4eii. Historic autoclave*

After the autoclave procedure was run, there were no visual differences in the glazes. The only noticeable change was that the pieces, despite being slightly wet (from the steam application), looked slightly brighter than they had before the procedure. This change was minimal, and due to its subjective nature, not measured in any way.

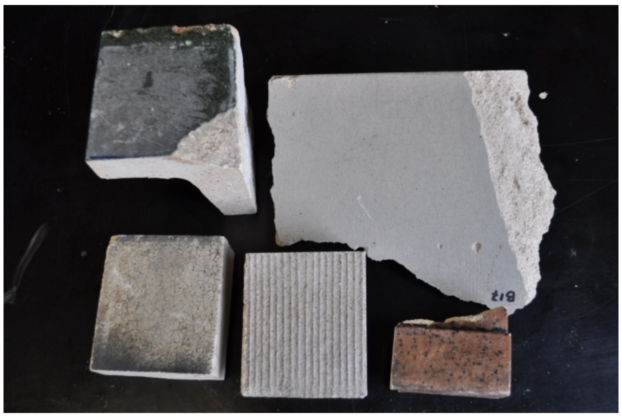


Figure 86: Samples (one per group) after undergoing the autoclave procedure

It is also important to take into account the difference in pressure that was prescribed by the historic test and what was available. A difference of 130 psi is significant, and therefore, this test was not an accurate representation of historic procedures.

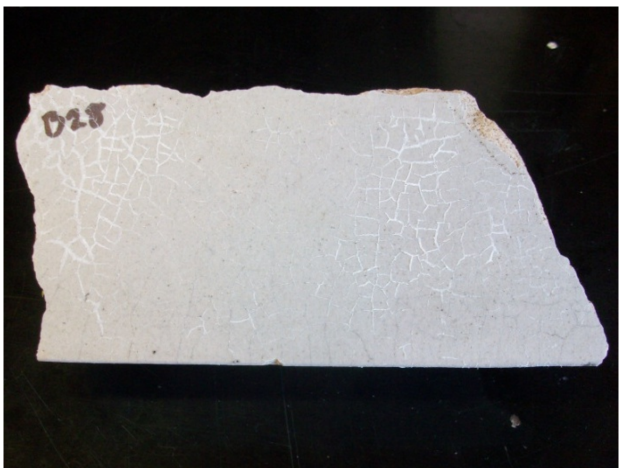
*4eiii. Freeze-thaw*

No visual changes were noticed on any samples subjected to freeze-thaw cycles. However, the air flow created by the fan in the freezer caused the water to freeze at uneven heights; some samples were standing in virtually no ice, others in 1 ½ inches. Because pieces were moved daily, this did not warrant extreme concern.

After 24 cycles, a small piece of clay body from sample B19 broke loose. The piece was from the rear of the sample, with no glaze attached. No other samples exhibited such failure, partial or complete.

*4eiv. Crystallization*

A crazing pattern similar to that discussed in the boiling salt bath test was also seen in the BRE Crystallization test samples. After 3 days of testing, salt deposits lined the crazing lines of sample group B pieces. As with the previous test, these patterns were unevenly distributed over the glazed surfaces. Other similarities to the historic test included the concentration of salt deposits around exposed  clay body on broken glazed surfaces, and darker crazing lines on sample group A pieces.



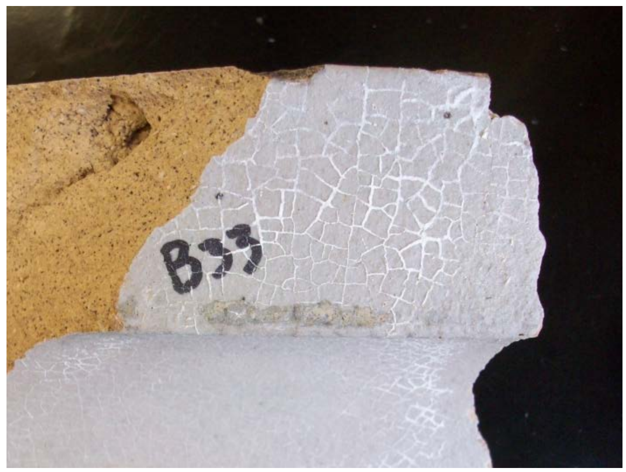




Figure 87 (left): Salt patterns over crazing lines on sample B28
Figure 88 (right): Salt patterns over crazing lines on flat and curved surfaces of sample B33

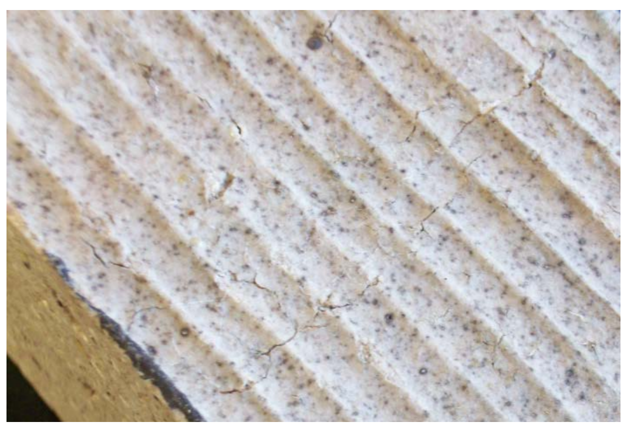


Figure 89 (left): Salt deposits around a broken glazed surface on sample B34
Figure 90 (right): Darkened crazing lines on sample from group A

After approximately 7 days of testing, some of the surfaces resting on the grille of the oven began to develop rust stains. Excess rust was brushed off the grille, but could not be completely removed. Although the glazed surfaces of terra cotta, upon which surface adhesion tests would be performed, were never resting on the rusted grille, it is unknown what effect the rust may have once samples were submerged in the sodium sulfate bath.

Upon testing completion, a layer of sediment similar to that seen in the historic boiling salt bath test could be seen in the bottom of the soaking container. As with the historic test, it is unknown if this was from disintegration caused by the weathering procedures, or simply previously loose material falling off the samples. It is also worth noting that as the samples were submersed in the sodium sulfate solution, bubbles always appeared on the surface of the water, presumably as the pieces became saturated.

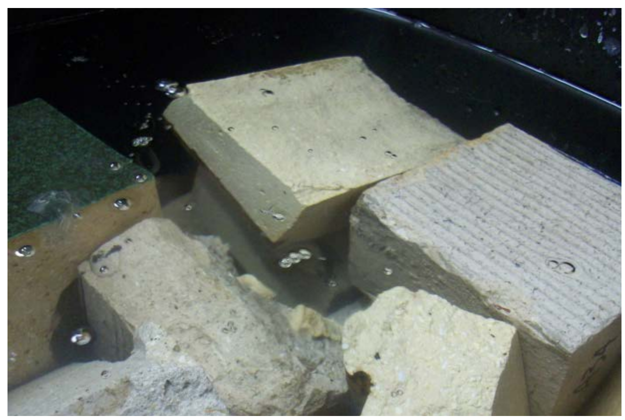


Figure 91 (left): Sediment remaining after test completion
Figure 92 (right): Bubbles rising from recently submerged samples

*4ev. Surface adhesion*

All but two samples achieved glaze failure under force from the Elcometer 106. Data from these tests is represented in the following table.

| | Boiling Salt Bath | | Autoclave | | Freeze-Thaw | | Crystallization | | Control | |
|---|---|---|---|---|---|---|---|---|---|---|
| | A1 | 500 | A10 | 800 | A19 | 825 | A28 | ♦ 450 | A37 | 900 |
| | A2 | 550 | A11 | 750 | A20 | 375 | A29 | 650 | A38 | 550 |
| | A3 | 675 | A12 | Δ ♦ 1000 | A21 | 450 | A30 | 625 | A39 | ♦ 550 |
| | A4 | 625 | A13 | Δ 1000 | A22 | 450 | A31 | ‡ 525 | A40 | ♦ 900 |
| | A5 | ♦ 500 | A14 | 1000 | A23 | ♦ 625 | A32 | 650 | A41 | 750 |
| | A6 | Δ 1000 | A15 | 525 | A24 | 475 | A33 | Δ 1000 | A42 | 700 |
| | A7 | 775 | A16 | 575 | A25 | † 400 | A34 | 750 | A43 | 1000 |
| | A8 | 450 | A17 | Δ 1000 | A26 | ♦ 425 | A35 | 300 | A44 | 500 |
| | A9 | 450 | A18 | 500 | A27 | 425 | A36 | † 200 | A45 | 475 |
| **Average** | | 614 | | 794 | | 494 | | 572 | | 703 |
| **Effectiveness (-)** | | 89 | | -92 | | 208 | | 131 | | |
| **Effectiveness (%)** | | 13% | | -13% | | 30% | | 19% | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | B1 | 775 | B10 | ♦ ■ 275 | B19 | 500 | B28 | 450 | B37 | 425 |
| | B2 | 325 | B11 | 550 | B20 | 550 | B29 | 300 | B38 | 275 |
| | B3 | 525 | B12 | 400 | B21 | 500 | B30 | 450 | B39 | 600 |
| | B4 | 350 | B13 | 450 | B22 | 500 | B31 | 475 | B40 | 375 |
| | B5 | 350 | B14 | 550 | B23 | 800 | B32 | 400 | B41 | 550 |
| | B6 | 675 | B15 | 500 | B24 | 550 | B33 | 325 | B42 | 300 |
| | B7 | 450 | B16 | 525 | B25 | 400 | B34 | ♦ 650 | B43 | 275 |
| | B8 | 350 | B17 | ♦ 400 | B26 | 875 | B35 | ♦ 450 | B44 | ♦ 275 |
| | B9 | 350 | B18 | 700 | B27 | 275 | B36 | 650 | B45 | 475 |
| **Average** | | 461 | | 483 | | 550 | | 461 | | 394 |
| **Effectiveness (-)** | | -67 | | -89 | | -156 | | -67 | | |
| **Effectiveness (%)** | | -17% | | -23% | | -39% | | -17% | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | C1 | 425 | C4 | 400 | C7 | ◊ | C10 | 400 | C13 | ◊ |
| | C2 | 525 | C5 | 500 | C8 | † 375 | C11 | 475 | C14 | † 775 |
| | C3 | 525 | C6 | 900 | C9 | ♦ 400 | C12 | 650 | C15 | 550 |
| **Average** | | 492 | | 600 | | 388 | | 508 | | 663 |
| **Effectiveness (-)** | | 171 | | 63 | | 275 | | 154 | | |
| **Effectiveness (%)** | | 26% | | 9% | | 42% | | 23% | | |

| | Boiling Salt Bath | | Autoclave | | Freeze-Thaw | | Crystallization | | Control | |
|---|---|---|---|---|---|---|---|---|---|---|
| | D1 | 650 | D3 | 600 | D5 | 750 | D7 | 525 | D9 | 500 |
| | D2 | 550 | D4 | 500 | D6 | 400 | D8 | □ 600 | D10 | 700 |
| **Average** | | 600 | | 550 | | 575 | | 563 | | 600 |
| **Effectiveness (-)** | | 0 | | 50 | | 25 | | 38 | | |
| **Effectiveness (%)** | | 0% | | 8% | | 4% | | 6% | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | E1 | 300 | E4 | 375 | E7 | 325 | E10 | 450 | E13 | 650 |
| | E2 | 625 | E5 | 375 | E8 | 425 | E11 | 500 | E14 | 450 |
| | E3 | 325 | E6 | 475 | E9 | 300 | E12 | 400 | | |
| **Average** | | 417 | | 408 | | 350 | | 450 | | 550 |
| **Effectiveness (-)** | | 133 | | 142 | | 200 | | 100 | | |
| **Effectiveness (%)** | | 24% | | 26% | | 36% | | 18% | | |

♦ No bearing ring used.

‡ Force exerted by Elcometer cracked sample, in addition to glaze and clay body failure at dolly location.

Δ Sample required sustained pressure at 1000 psi to create failure.

◊ Epoxy failed before terra cotta sample- no data. Likely due to improper epoxy adhesion to dirty/salty surface.

† Failure only over part of the dolly surface area.

■ Void revealed under testing location, likely contributed to low force readout.

□ Glaze failure only, no clay body removed. Indication of poor glaze-clay body adhesion.

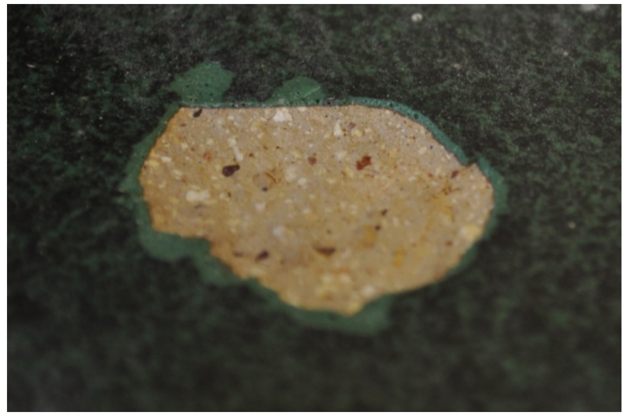


Figure 93 (left): Sample A31 cracked during testing
Figure 94 (right): Visible cross-section of glaze on sample C9

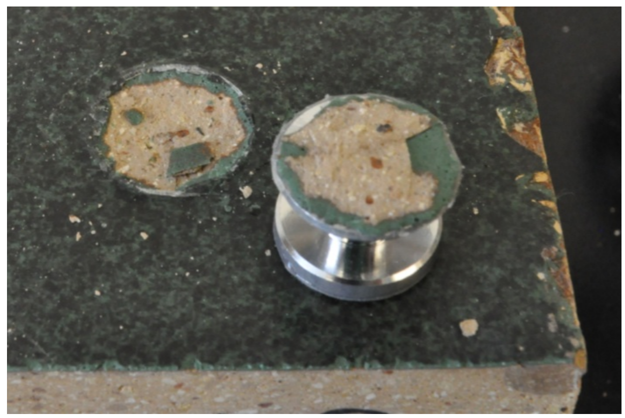



Figure 95 (left): Typical pull test result, as seen on sample E12
Figure 96 (right): Typical pull test result, as seen on sample C12

Figure 97 (left): Partial failure on sample A11
Figure 98 (right): Partial failure on sample C14

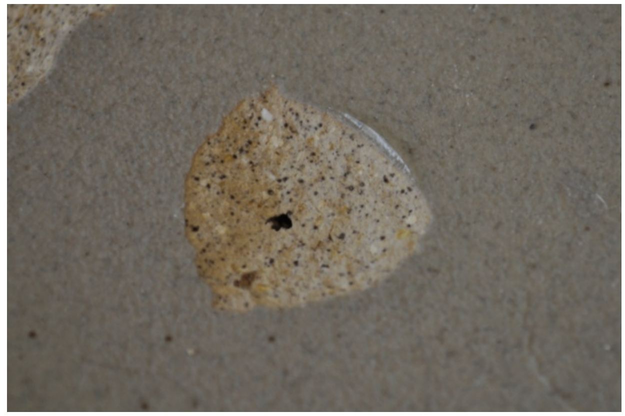



Figure 99 (left): Failed pull test on sample C13
Figure 100 (right): Void revealed under pull test location on sample B10

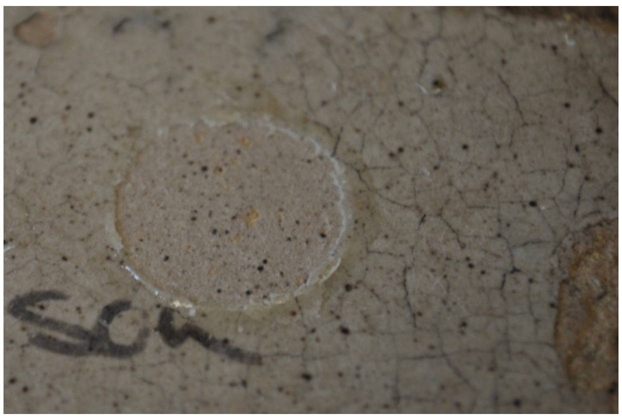


Figure 101: Glaze failure without clay body failure on sample D8

## 5. RESULTS & INTERPRETATION

This thesis has proved that artificial weathering is not a purely modern concept, has shed light on the variety of tests conducted, and also the range of methods used to determine their effectiveness. Historically, the effect of weathering was noted by visual changes to the unit (such as flaking, cracking, or crazing), decreased compressive strength, or increased absorption rates. Contemporarily, value is measured by major cracking, or, more commonly, a loss in weight of the specimen, which indicates decay.

Artificial weathering techniques are not dependent on a glazed surface; tests may, and often are, performed on unglazed units. Tests are designed to weaken the unit as a whole, and thus, in theory, deteriorate both the clay body and the glaze-clay interface.

However, since this thesis focused on glaze fit and defects, and the relationship of the glaze to the clay body, the evaluation method for these tests needed to specifically measure this relationship, not overall body weakening. It was for this reason that the surface adhesion pull test was chosen to evaluate all weathering methods employed. This allowed for consistent results between tests, and a specific applicability to the topics researched in the historic literature survey.

The results obtained from the pull tests conducted, however, were scattered. It was assumed that the highest values would always come from the control groups, and that various weathering methods would subsequently lower these values. This was often not the case, especially in samples from group B, where the average value for the control group was lower than all of the weathering tests. In sample groups A, C, and E, the freeze-thaw test had the greatest weakening impact on the samples, but due to the wide range of data (375-825 psi in group A), this trend cannot be conclusively verified. Similarly, the results appear to indicate that both salt bath tests had similar effects, given the close range of effectiveness (as measured as a percentage) within each sample group, but it must also be noted that these ranges were often broad: the sample A crystallization group produced data ranging from 200-1,000 psi. This being the case, it is difficult to speak to the efficacy of these artificial weathering tests in terms of facts and numbers.

This variation within all of the sample groups is likely due to the inconsistencies inherent in terra cotta as a material. Even if a single production batch was made with the same materials and processes, there is still room for variation: inconsistent pressings and finishing, position in the kiln, handling, position on the building, and varieties in the natural materials used. This thesis proved that even when testing 9 samples from the same building in a single test group, the results may still be widely inconsistent. This inherent variability of terra cotta made it difficult to draw conclusive results in these experiments.

## 6. RECOMMENDATIONS & CONCLUSIONS

Due to logistical and scheduling constraints, the full extent of historic terra cotta testing methods was not researched. There are more sources that are surely worth chronicling, should this research be continued by a future student or historian. These sources include:

- Transactions of the American Ceramic Society
- The Bulletin of the American Ceramic Society
- The Clay-worker
- Brickbuilder
- Brick and Clay Record

If such studies were carried out, a similar testing protocol to the one used in this thesis could be followed. However, due to the discrepancies found after conducting surface adhesion pull tests, this testing program should not be carried out with historic material. Rather, the use of newly manufactured samples would decrease the chance for variability between samples- although it must be recognized that this can never be completely eliminated. Therefore, I would suggest testing at least 10 samples per group per test, and would urge future researchers to only use new material as a means of evaluating and comparing weathering methodologies. It would also be preferable to allow the freeze-thaw test to run for the recommended full 50 day cycle, to allow for optimal and most accurate results, and to locate an autoclave capable of producing 150 psi, as was prescribed by historic literature. The ability to only produce 20 psi in this thesis severely lessened the impact this procedure may have been able to impart on the samples, and as such it cannot be viewed as an accurate reproduction of a historic test.

Although not directly related to artificial weathering, both salt bath tests (the historic boiling test and the contemporary BRE test) were found to be good indicators of crazing on a sample when salt deposits outlined these small cracks. In one instance, in group A, the tests were useful in identifying defects that had not been noticed without the use of a microscope. The results from the boiling salt bath showed more visibly dramatic results within a shorter time frame, and this test could potentially be developed by future conservators as a test for identifying glaze defects.

Despite these limitations, the information gained from the *Journal of the American Ceramic Society* was valuable. The historic literature survey revealed the depth and breadth of issues related to terra cotta durability that ceramicists of the early 20$^{th}$ century were considering. Their testing methodologies often showed sophistication and a thorough understanding of the workings and development of architectural terra cotta. Many ideas employed nearly a century ago are still in use today, and there could be further applications for processes that have been, up until now, forgotten. These include the salt bath and autoclave tests, but further research with suitable samples is needed to determine the efficacy of these tests and any possible usefulness they may be able to lend to the future of the profession.

As the field continues to evolve with technological developments and better understandings of material behavior, it is important to remember terra cotta's past. Many historic techniques may still have an application in today's industry of construction and conservation, and we must continue to strive to learn from those craftsmen that came before us.

## APPENDIX: GLOSSARY

**Cones-** Cones are used to determine the heat of a kiln. Small, cone-shaped pieces of clay are placed in the kiln, and each is calibrated to bend at a different temperature level. These denote firing temperatures- a unit may be said to be fired to “cone 6,” which would correlate to a certain degree of heat.

**Cornwall stone-** “A partially decomposed granite containing feldspar, quartz, kaolinite, mica, and trace amounts of fluorspar. It has a variable chemical consistency, with properties similar to feldspar. It is commonly used in…glazes as a flux, helping to bring other materials into a melt.” (Hammill & Gillespie, *Cornwall Stone Fact Sheet*)

**Crawling-** “A parting and contraction of the glaze on the surface of ceramic ware during drying or firing, resulting in unglazed areas bordered by coalesced glaze.” (ASTM C242-01, p. 3) It is caused by tension left in the glaze after fusion with the clay body. (Hewitt, “Monograph and Bibliography on Terra Cotta”)

**Crazing-** “The cracking that occurs in fired glazes or other ceramic coatings as a result of tensile stresses.” (ASTM C242-01, p. 3) The cracks can be on the surface of the glaze, throughout the whole thickness of the glaze, or even extend into the clay body. (Hewitt, “Monograph and Bibliography on Terra Cotta”)

**Dunting-** “The cracking that occurs in fired ceramic bodies as a result of thermally induced stresses.” (ASTM C242-01, p. 4)

**Efflorescence-** A “crystalline deposit, usually white, of water-soluble compounds on the surface of masonry.” (ASTM C1232, p. 2)

**Engobe-** “A slip coating applied to a ceramic body for imparting color, opacity, or other characteristics, and subsequently covered with a glaze.” (ASTM C242-01, p. 4) “A term of wide meaning, often interchangeable with slip but including other materials. An engobe is used to cover a clay, produce a bugger layer and give a different surface, texture, and colour. It is applied by brush, dip, spray, etc. Clay slips and glaze slips are essentially fluid in forms. An engobe can be in a jelly or stiff form. It is often half-way between a clay and a glaze in composition and contains materials which are normally considered glaze materials. It therefore fires to a more vitreous state than the body which it covers. However, since it does not fuse to a glassy state, it cannot be called a glaze. And since it can be composed entirely of non-clay materials, it cannot be called a slip. The term engobe is often the only one that is suitable." (Hamer, p. 126)

**Feldspar-** "A mineral aggregate consisting chiefly of microcline, albite, or anorthite or combination thereof." (ASTM C242-01, p. 4)

**Firecrack-** "The sharp, hair-line cracks that may appear on terra cotta under certain conditions are termed firecracks, cooling cracks, or dunting. They are caused by contraction strains being set up in the body during the cooling. If these strains are severe the piece may be cracked when drawn from the kiln. If the strains are less severe, the cracking may not occur until a considerable time after the piece has been set in the building." (Hill, "Some Experiments on the Firecracking of Terra Cotta")

**Flux-** "A substance that promotes fusion in a given ceramic mixture." (ASTM C242-01, p. 5)

**Fusibility-** Historic literature was not specific about the methods used to test fusibility, whether it was examined within the glaze only, or between the glaze and the clay body. However, the implied meaning is the ability of the glaze to melt, creating a near-liquid material, that attaches uniformly to the clay body without separation.

**Geode-** "Small hollow bodies with the interior face of the cavity lined with small crystals….2 to 3 millimeters in diameter." (Parmelee, "An Unusual Cause of Spalling of Sewer Pipe")

**Glaze-** “A ceramic coating matured to the glassy state on a formed ceramic article, or the material or mixture from which the coating is made.” (ASTM C242-01, p. 5)

**Bright glaze-** “A colorless or colored ceramic glaze having high gloss.”

**Bristol glaze-** A glaze containing feldspar, flint, clay, whiting, and zinc oxide. (E.C. Hill. “Some Data on the Development of Terra Cotta Glazes”)

**Clear glaze-** “A colorless or colored transparent ceramic glaze.” (ASTM C242-01, p. 5)

**Crystalline glaze-** “A glaze containing macroscopic crystals.”

**Fritted glaze-** “A glaze in which a part or all of the fluxing constituents are prefused.”

**Mat (matte) glaze-** “A colorless or colored ceramic glaze having low gloss.”

**Opaque glaze-** “A nontransparent colored or colorless glaze.”

**Raw glaze-** “A glaze compounded primarily from raw constituents, that is, containing no prefused materials.” (ASTM C242-01, p. 5)

**Salt Glaze-** “A glaze produced by the reaction, at elevated temperature, between the ceramic body surface and salt fumes produced in the kiln atmosphere.” (ASTM C242-01, p. 8)

**Semi-mat glaze-** “A colorless or colored glaze having moderate gloss.”

**Slip glaze-** “A glaze consisting primarily of a readily fusible clay or silt.”

**Vellum glaze-** “A semi-mat glaze having a satin-like appearance.” (ASTM C242-01, p. 5)

**Open-burning-** A clay that, when fired, is porous. *Antonym:* **Vitrifying**

**Plastic-** “A descriptive term applied to a material that exhibits the property of plasticity or stickiness, where plasticity is the ability of a material to undergo substantial deformation without fracturing.” (ASTM C242-01, p. 7)

**Peeling-** *see shivering*

**Refractoriness-** The ability of terra cotta “to carry [a] load without warpage or deformation.” (Allen, “Better Terra Cotta Slabs”)

**Sagger-** A fired, box-like piece of terra cotta within which a unit to be fired is placed. The sagger shields the new unit from kiln flames and debris. The term “sagger” may be derived from the word “safeguard.” (http://www.encyclopedia.com/doc/1O27-saggar.html)

**Scum-** *see efflorescence*

**Shivering-** “The splintering that occurs in fired glazes or other ceramic coatings as a result of critical compressive stresses.” (ASTM C242-01, p. 9)

**Slab-** Although the February 1925 article about slabs does not give an explicit definition, slabs are understood to be a surface upon which wares rested in the kiln; slabs were subjected to many firings as they supported other units, probably in a manner similar to saggers.

**Slag-** “The non-metallic product consisting essentially of silicates and alumino silicates of calcium and other bases, that is developed in a molten condition simultaneously with iron in a blast furnace.” (ASTM C125)

> **Furnace Slag-** “Formed when iron ore or iron pellets, coke and a flux (either limestone or dolomite) are melted together in a blast furnace. When the metallurgical smelting process is complete, the lime in the flux has been chemically combined with the aluminates and silicates of the ore and coke ash to form a non-metallic product called blast furnace slag.

During the period of cooling and hardening from its molten state, blast furnace slag can be cooled in several ways to form any of several types of blast furnace slag products," including granulated slag. (www.nationalslag.org)

**Granulated Slag-** Slag that is "rapidly cooled by large quantities of water to produce a sand-like granule that is primarily ground into a cement." (www.nationalslag.org)

**Slip Coating-** "A ceramic material or mixture other than a glaze, applied to a ceramic body and fired to the maturity required to develop specified characteristics." (ASTM C242-01, p. 9)

**Spall-** A separation of glaze from the clay body, usually with a small amount of clay body attached, due to internal pressure exerted on the glazed surface.

**Vitrification-** "The progressive reduction and elimination of porosity of a ceramic composition, with the formation of a glass phase, as a result of heat treatment." (ASTM C242-01, p. 10)

**Vitrifying-** A clay that, when fired, is dense and glass-like; a clay that has reached vitrification. *Antonym:* **Open-burning**

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